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THE ANALYST.

JANUARY, 1902.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

THE monthly meeting of the Society was held on Wednesday evening, December 11, in the Chemical Society's Rooms, Burlington House. The President (Dr. J. AUGUSTUS VOELCKER, M.A., B.Sc.) occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. Horatio Ballantyne, F.I.C., M. Hunter, M.A. (Oxon.), Ernest Alfred Lewis, Frederick J. Lloyd, F.I.C., Albert E. Parkes, F.I.C., and Thomas Tickle, B.Sc. (Lond.), were read for the second time; and a certificate in favour of Dr. L. T. Thorne, F.I.C., Technical Chemist, 8, Dynevor Road, Richmond, Surrey, was read for the first time.

Messrs. L. Kidgell Boseley and Martin Priest were appointed to act as auditors of the Society's accounts for the year 1901.

The following papers were read: "A Contribution to a Knowledge of the Chemistry of Cider," by Alfred H. Allen; "On the Mannitic Fermentation of Wine," by Philip Schidrowitz, Ph.D.; "On Cocoa Extracts," by E. G. Clayton; and a "Note on Commercial Zinc White and other Pigments," by E. G. Clayton.

THE DETERMINATION OF CARBON IN STEEL BY DIRECT COMBUSTION.

BY BERTRAM BLOUNT, F.I.C.

(*Read at the Meeting, November* 6, 1901.)

IN the ANALYST for June, 1900, I have discussed the question of devising a practicable method for the determination of carbon in steel by direct combustion—*i.e.*, without having recourse to the usual process of previously removing the iron by solution in potassium cupric chloride or the like solvent. I would lay emphasis on the adjective "practicable." There are many prescriptions for burning steel and collecting the carbon dioxide representing its carbon, but they hardly satisfy the condition of being workable. Either the metal has to be finely divided by mechanical means or the iron must be removed by volatilization in a current of chlorine or bromine without attacking the carbon, which is only possible if air and moisture be completely excluded—a condition difficult of attainment. Such operations fall far short in simplicity and accuracy of a method which shall be capable of oxidizing steel directly

and completely in substantial fragments, such as drillings or turnings, and of yielding the whole of its carbon as carbon dioxide free from other gases absorbable by potash.

In the paper already referred to, and in another on the determination of oxygen in commercial copper (ANALYST, xxi., 57), I have pointed out that it is unnecessary for the chemist in performing analytical operations of this class to restrict himself to the use of temperatures commonly attained in the combustion furnace. With proper precautions a temperature of 1,100° C. is as manageable as is one of 800° C., and reactions which are slow and imperfect at the latter take place smoothly and completely at the former. Such difficulties as have to be overcome arise from the fact that ordinary apparatus and appliances are not as a rule designed for the production and utilization of these higher temperatures, and certain modifications in instruments and procedure are called for.

In my previous experiments I endeavoured to burn steel turnings in oxygen without admixture of any oxidizing or fluxing agent. I found that there was a considerable tendency to leave a core of unburnt metal which was usually free from carbon, but, if occurring, raised a doubt whether the combustion was complete; if heating was continued until this core was oxidized, the operation was unduly prolonged, and errors inseparable from a lengthy combustion crept in. In spite of these obstacles, the results obtained were sufficiently encouraging to warrant further study of the subject, and I turned my attention to the use of some suitable oxidant. Of all the oxidizing materials which suggested themselves, lead chromate appeared preferable, because, unlike lead oxide (no doubt an ideal carrier and flux for the purpose in hand), it does not attack porcelain violently, and, further, when temporarily reduced, as it may be by the steel even in an atmosphere of oxygen, it does not yield fluid masses of metallic lead, and consequently does not imperil the integrity of platinum. Therefore two standard refractory materials become available.

Using lead chromate, the process resolved itself into this: A weighed quantity of the steel, usually 5 grammes, in the form of drillings or turnings, often of relatively stout section, was mixed with 15 to 20 grammes of lead chromate which had been recently fused and thus freed from oxidizable impurities. The mixture was put into a large porcelain boat, the boat was placed in a long, deep tray of platinum sheet, and the whole slid into a porcelain tube. The object of the platinum tray was to prevent any chromate which might creep from coming in contact with the porcelain tube. It was found impracticable to use a platinum boat, as the hard fused mass of chromate and oxide of iron left after combustion was difficult to remove without injuring the boat. The front part of the porcelain tube contained copper oxide, and was heated in a Fletcher combustion furnace. The back part was similarly heated, but the precise spot where the boat lay was raised to a temperature considerably higher than could be reached by the combustion furnace. After many trials of different burners, a benzoline blast-lamp was found to be fairly effective for this purpose. I may remark in passing that there is an opening for a burner as handy as a common bunsen, but giving the temperatures usually requiring a blast for their production—*i.e.*, ranging from the melting-point of copper to that of cast iron. I have not yet been able to procure a burner of this description from chemical dealers, and in many operations am compelled to have recourse to a mechanical blower, when a self-contained blast-

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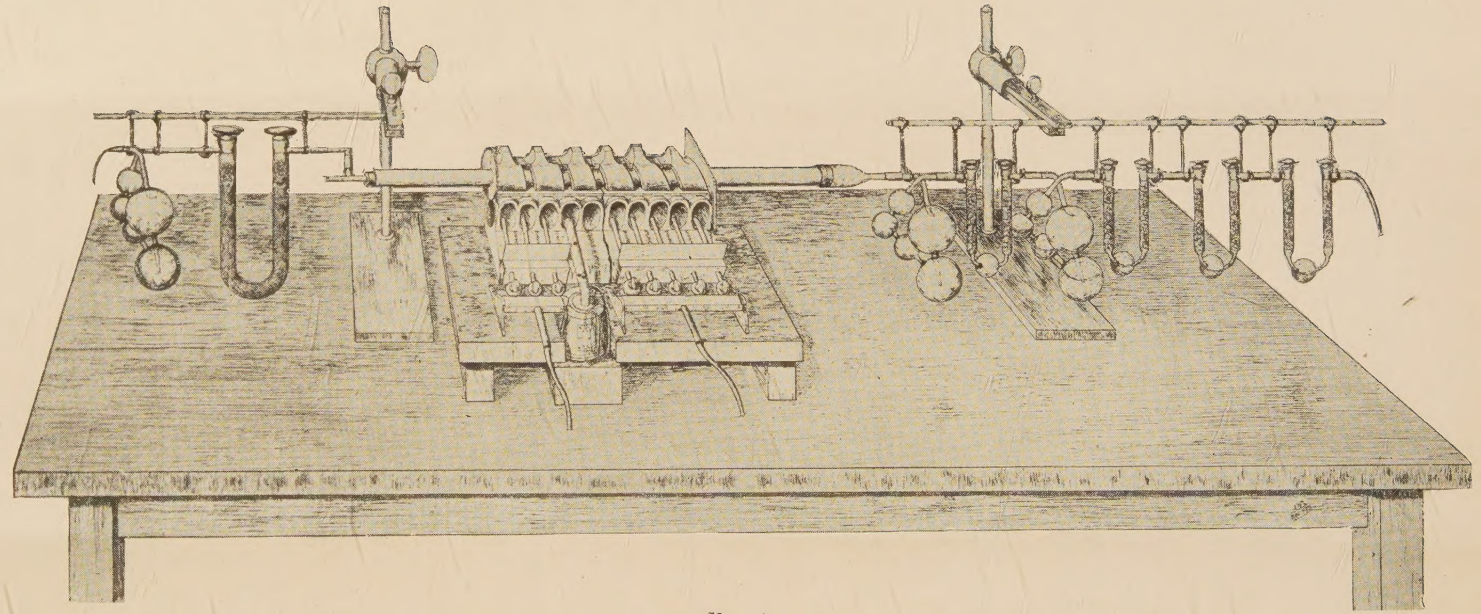


FIG. 1.

lamp of ample power would be preferable in every way. An electrical method of heating may be the best solution, but there is nothing of the kind in common use except little muffles for dental work, and I have not had time to design such an apparatus for my special needs.

The arrangement of the whole apparatus is shown in the accompanying diagram (Fig. 1).

Starting on the left, we have a potash bulb and a U-tube of soda-lime to purify the oxygen, and afterwards the air used for sweeping out the tube. The glass T-piece with free flattened end allows the boat to be seen in the tube during combustion. A Fletcher combustion furnace is used, supplemented by a benzoline blast-lamp at the point where the boat is placed. Connection at the far end of the porcelain tube is made by a glass cap on a rubber ring instead of the usual cork. This cap is drawn down so as to fit small tubing for connection with the absorbing train. The first potash bulb is empty, and serves merely for cooling. It can, if necessary, be

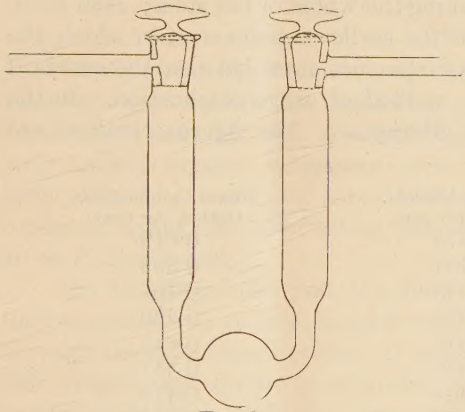


FIG. 2.

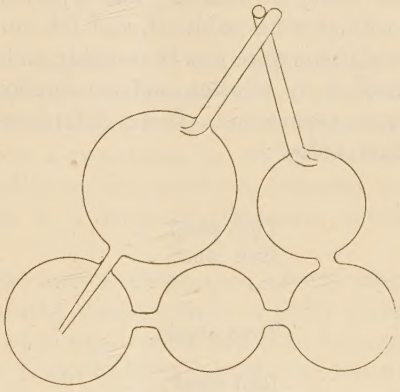


FIG. 3.

immersed in a beaker of water. A U-tube of sulphuric acid on pumice dries the gas, and a potash bulb and similar U-tube constitute the weighed absorption apparatus. A second weighed sulphuric acid U-tube serves as a check on the efficiency of the first. It is weighed every three or four combustions, and should gain only fractions of a milligramme. A guard tube of sulphuric acid on pumice completes the train, and to this the pump can be attached.

The U-tubes are designed so that the gas is compelled, not merely to traverse two columns of pumice, but to bubble through the acid. The bottom bulb forms a useful store of acid with which the pumice can be refreshed, for it often happens that when sulphuric acid on pumice alone is used, the acid on the surface of each piece may be diluted and exhausted, although there is still plenty of strong acid in the centre of each piece of pumice. The tube will then cease to act efficiently, but if it contains a store of acid actually fluid, this can be washed over the pumice in each limb, and replace the used-up acid. A diagram of the tube is shown (Fig. 2).

The potash bulb designed by my chief assistant, Mr. S. Dickson, is also novel, and an improvement on the ordinary forms. A diagrammatic sketch is given

(Fig. 3). The potash bulb differs from most apparatus of the kind by being asymmetrical. The entrance limb is larger and heavier, and the bulb hangs naturally in its proper position for absorption, so that no tying up or tilting is necessary. An extra passage through the liquid is secured by an internal tube in the first bulb, and both entrance and exit tubes project a little way into their respective bulbs, and are turned aside from the centre, their ends being also contracted. Under reasonable conditions of use splashing is almost impossible. I use these bulbs for all absorption work, and find them preferable to any other design with which I am acquainted.

When the method was carried out in this manner, satisfactory results were obtained. The high temperature employed made it necessary to insure that the drying tubes were kept in excellent condition, and were not run too long without renewal.* It was also requisite to have a good length of tube projecting beyond the furnace, to avoid over-heating the rubber connections. A porcelain tube 26 inches by $1\frac{1}{8}$ inches was found convenient. With an adequate temperature and time of heating (about one hour), no difficulty was experienced in burning the whole of the steel; even thick turnings were oxidized, and left no core. In the earlier experiments, in which the conditions were less thoroughly understood, discrepancies occur between the standard method by solution and combustion and the method of direct combustion. In the later experiments these differences tend to disappear. The figures obtained are given below:

	Solution Method. Carbon per cent.	Direct Combustion. Carbon per cent.
Tyre steel	0·675	0·619
Rail steel	0·404	0·393
,, ,, ...	0·399	0·407
,, ,, ...	0·378	0·330
Tyre steel	0·643	0·645
,, ,, ...	0·618	0·557
Rail steel	0·401	0·419
,, ,, ...	0·432	0·435
,, ,, ...	0·430	0·408
,, ,, ...	0·457	(1) 0·422
		(2) 0·393
		(3) 0·438
Rail steel	0·470	0·429
Nickel steel	0·323	(1) 0·324
		(2) 0·324
Tyre steel	0·459	Improved apparatus for cooling and absorption 0·445
Structural steel (plate) ...	0·288	0·277
Rail steel	0·455	0·467
,, ,, ...	0·568	0·575

It is evident from these results that the method is reliable when properly handled. But it still has several defects, due chiefly to the imperfectly refractory nature of the materials at one's command. At the temperature necessary the glaze of royal Berlin porcelain is slightly sticky, and the platinum tray adheres thereto.

* The efficiency of the train of absorption tubes finally adopted was tested by numerous blank experiments, and by the use of a second drying tube after the potash bulb weighed independently of the drying tube proper.

It is difficult to detach, and when forcibly removed is itself often deformed and injured, and removes fragments of the glaze, frequently leading to the breakage of the tube. The porcelain boat also adheres to the platinum tray, and sometimes can hardly be withdrawn from the tube without tearing the platinum, which is itself stuck to the porcelain tube. Various devices suggest themselves to remedy these inconveniences, and these I propose to examine when work allows. In the meantime, I am of opinion that the method will suffice to check the results obtained by the usual process in the event of a doubt arising. So much has the customary process been elaborated that there are schools of analysts who quarrel about the position of a potash bulb, and seek to standardize every piece of apparatus. All this is, of course, mischievous and absurd, and it is well to remember that the real object in view is to determine accurately the percentage of carbon irrespective of any particular procedure. Hence a process which has not yet suffered from pseudo-standardization presents some advantages.

Discussion.

Mr. Raymond Ross inquired whether it would be possible to use a piece of glass tube for each experiment instead of a permanent porcelain tube with boat and platinum shield. He had himself obtained very satisfactory results in carrying out with certain organic substances some combustion experiments, in which he had been obliged to use a very short furnace, by employing lead chromate alone as an oxidizing agent and conducting the combustion in a piece of glass tube about 10 or 12 inches long.

Mr. Archbutt said that Mr. Blount was to be congratulated upon having, after long perseverance, at last found a method of burning steel perfectly without going through the roundabout process of decomposing with copper salts, and then burning and weighing the carbon so separated. Of course, it was to be hoped that Mr. Blount would be able ultimately so to improve the process that it would be sufficiently rapid to replace the ordinary more cumbrous method. At present it would hardly do that, for in the ordinary process a number of samples of steel would be dissolved at the same time in the double chloride of copper and potassium, and the carbonaceous residues filtered off one after another; and, as the combustion took place fairly quickly and at a comparatively low temperature, a considerable number of determinations could be carried out in one apparatus in a single day. The new method, however, as he had remarked on a previous occasion, was valuable in its present stage because it afforded a complete check upon the results of the ordinary process. Mr. Blount, in referring to the use of lead chromate, had used the text-book expression "recently fused." Surely, however, it was immaterial whether the fusion had taken place recently or not, provided that the lead chromate really had been fused and had been so kept that it could not absorb moisture or carbonaceous matter. In the course of some experiments in standardizing a pyrometer for obtaining the cooling curves of metals, he had found it possible with a small furnace, by using compressed air at about 70 pounds per square inch and coal gas under a pressure of 6 or 7 inches of water, to obtain temperatures very much higher than with gas at ordinary pressure and air supplied from a blower. He had easily melted copper in

this way. Another suggestion was that a certain proportion of oxygen might be mixed with the air from the blower, by which a higher temperature would be obtained without increase of pressure. He would like to hear at what rate the oxygen was passed over the burning steel, because, from what was said about the high temperature of the gas that had to be dried, it would seem that the oxygen must be passed through at a very rapid rate. At a moderate rate—say from four to six bubbles per second—the gas might be expected to become sufficiently cool by radiation very soon after escaping from the combustion tube. If the rate were more rapid, possibly soda-lime might be a better absorbent than caustic potash for the carbonic acid. He had at one time experienced some difficulty in the absorption of the carbonic acid by potash and the weighing of the potash bulbs. His custom was not to aspirate air through the combustion furnace after burning the carbon, but first of all to pass oxygen through the apparatus and weigh the bulbs full of oxygen at intervals of ten minutes until constant, then burning the carbon and weighing the bulbs again full of oxygen at similar intervals till constant. He had found, however, that the bulbs showed a tendency to lose a few tenths of a milligramme continuously, which was ultimately traced to the not quite sufficiently hygroscopic character of the drying tube at the end. In that tube had been used, not calcium chloride—a very imperfect drying agent—but broken-up pieces of ordinary stick-potash, which was very commonly used for the purpose. Dittmar, however, in his researches on the combining proportions of oxygen and hydrogen, had demonstrated the remarkable drying powers of potash, which had been freshly fused until it contained no water. So-called pure potash, when heated in a platinum or a nickel dish, lost from 15 to 20 per cent. of water, and became perfectly anhydrous; and if this, after cooling, were broken into small pieces and used for filling the small tube at the end, no loss occurred in the potash bulbs within ten minutes or a quarter of an hour, and they could be got almost absolutely constant. He thought that if Mr. Blount were to give up aspirating air through the apparatus, and weigh the bulbs full of oxygen instead, the results would be obtained perhaps a little more quickly.

Dr. Dyer inquired whether the use of sulphuric acid for drying purposes was wholly desirable in view of the fact, which had been pointed out by Mr. Allen in the course of the discussion on Mr. Blount's previous paper on the same subject, that sulphuric acid was liable to absorb small quantities of carbonic acid.

Mr. Blount said he was afraid it would not be possible to use a glass tube as suggested by Mr. Ross, because the essence of the process was the high temperature employed. The temperature necessary was so considerable that the glass would blow out with the ordinary pressure needed to drive the oxygen through the train of apparatus. Even when chromate of lead was used, the temperature requisite in order completely to burn steel which was in stout fragments was such that the use of glass, however hard, was impracticable. He acknowledged freely that at present he had no intention of putting this method forward as capable of replacing the ordinary process. He was alive to the necessity of speed, and the method might not compete in effective speed with the older process; it was true that its real speed was greater, because, although the combustion took longer, there was no necessity for dissolving and filtering. He thought he might claim for it that it could be

employed instead of the old process in any case of difficulty or doubt as to the purity of the reagents used, or when it was desired to fall back upon a totally different method, which should not involve any single step at all comparable with those of the ordinary process. He agreed with Mr. Archbutt's comment as to the term "recently fused," and, in fact, had erased it once, but, the search for a more suitable expression having been unsuccessful, he had replaced it. Clearly it was merely essential that the lead chromate should be kept clean after fusion, though it was perhaps better practice that the fusion should be recent, so as to minimize any chance of dirt being present, and that was the only excuse for the term. He had tried the use of what was known as "press gas," which, at a pressure of a few inches, was no doubt comparable with the pressures mentioned by Mr. Archbutt; then moderate pressure was hardly enough, but if gas under a pressure of a few pounds were available, it would probably not be necessary to use a benzoline blast-lamp. There was no difficulty in absorbing the carbonic acid when the oxygen was passed through at any reasonable rate. The trouble arose in absorbing the water. The use of fused potash might well be of assistance in this connection.

Mr. ARCHBUTT inquired where all the water came from.

Mr. BLOUNT said that a tangible quantity would come from the tube and apparatus, which could never be got completely dry, and in any case frequently had to stand overnight. Then, again, the copper oxide was somewhat hygroscopic, and there were a number of other sources of water which had to be reckoned with. He had never had occasion to try whether carbonic acid was absorbed by sulphuric acid, but that matter did not really bear upon the present case, because, although there might be some absorption if concentrated carbonic acid were passed over sulphuric acid, there would be none in the case of a gas containing but a small percentage of carbonic acid; one had to consider the partial pressure of the gas. These attenuated gases could be passed through even water without any appreciable loss of carbonic acid.

ARSENIC COMMITTEE.

The joint committee of the Society of Chemical Industry and of the Society of Public Analysts has now completed its investigations, and the report will be presented to the respective Societies in January.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOODS AND DRUGS ANALYSIS.

A Modification of the Sulphuric Acid Test for Formaldehyde in Milk. A. Gustav Luebert. (*Journ. Amer. Chem. Soc.*, xxiii., 682.)—The test is made as follows: Five grammes of coarsely-powdered potassium sulphate are placed in a 100 c.c. flask, 5 c.c. of the milk distributed over it by means of a pipette, and then 10 c.c. of concentrated sulphuric acid carefully poured down the side of the flask. The whole is allowed to stand, and if formaldehyde is present the crystals of potassium sulphate will acquire a violet colour in a few minutes, the liquid also gradually becoming coloured. If no formaldehyde is present, the liquid will at once turn brown, rapidly changing to black. The test is sensitive to a dilution of at least 1 part formaldehyde to 250,000 parts milk, and is given even more rapidly by milk which has stood for several hours than by a fresh sample. A. G. L.

Detection of Cocoanut Oil in True Butter. F. Ranwez. (*Ann. Pharm.*, 1901, vii., 241; through *Chem. Zeit. Rep.*, 1901, 240.)—Vandam has shown that the amount of acids, soluble in alcohol at 60°, contained in cocoanut oil, is much greater than that in true butter or in margarine. Expressed as c.c.s of decinormal potash, and calculated upon 5 grammes of the sample, five specimens of butter have given 10·3 to 11·1; cocoanut oil, 44·2; and margarine, 3·6. If the proportions of these acids insoluble in water, and those soluble in alcohol at 60°, are expressed in the same manner—*e.g.*, butter, 4·6 to 5·2; cocoanut oil, 42·0; margarine, 3·1, these differences become much larger. Therefore an addition of 12 or 13 per cent. of cocoanut oil to cow's butter would double the amount of water-insoluble acids in the alcohol-soluble acids, while an addition of 25 per cent. thereof would treble it. F. H. L.

The Determination of Starch in Cereals. L. Lindet. (*Journ. Pharm. Chim.*, 1901, xiv., 397-400.)—In order to obviate the difficulty of filtration in his method (ANALYST, xxii., 20), and to prevent cellular débris being weighed with the starch, the author proposes the following modification.

The starch washings passing through the sieve after the digestion with pepsin and hydrochloric acid are collected in a 500 c.c. conical flask, where the starch is allowed to subside, and is washed once or twice by decantation. As the starch settles but slowly, it is necessary to allow the liquid to stand from twelve to twenty-four hours after each washing, any bacterial action being prevented by the addition of a little formaldehyde.

The washed starch is then heated for an hour at 110° C. with 100 c.c. of 0·25, 0·5, or 1 per cent. sulphuric acid, and the resulting saccharified solution neutralized and diluted to 250 c.c. The amount of glucose and of dextrin are then determined

by reduction with Fehling's solution and polarization, and the original amount of starch calculated from the results.

When much oil is present in a cereal it is advisable to extract it prior to the digestion with pepsin, since otherwise the starch does not separate well in the decantation. C. A. M.

Some Analyses of Bulgarian Buffalo and Sheep Butter, Lard, and Walnut Oil. N. Petkow. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 826, 827.)—The maximum, minimum, and average results of the analyses of samples of buffalo and sheep butter are given below. It will be seen that the composition of these does not differ greatly from that of cow's butter, although the Reichert-Meissl figure for buffalo butter is somewhat higher. The average amount of water in the buffalo butters was 14·39 per cent. and in the sheep butters 12·72 per cent.

Buffalo Butter (14 samples).

	Specific Gravity at 100° C.	Refracto-meter Number at 40° C.	Reichert-Meissl Number.	Kött-storfer Number.	Iodine Number.	Insoluble Fatty Acids. Per Cent.	Free Acidity, ° Burstyn.
Maximum ...	0·8700	44·8	40·10	234·7	45·52	87·20	6·13
Minimum ...	0·8680	43·8	30·90	222·5	30·29	89·20	3·22
Average... ...	0·8692	44·2	34·20	229·0	36·75	88·19	4·51

Sheep Butter (12 samples).

Maximum ...	0·8695	45·5	29·37	234·0	38·48	89·90	3·21
Minimum ...	0·8690	42·5	23·21	223·0	29·70	87·90	0·50
Average... ...	0·8693	44·4	26·68	227·8	35·14	88·50	2·01

Lard (3 samples).

Maximum ...	0·861	52·0	0·52	198·8	65·56	96·14	1·08
Minimum ...	0·860	51·8	0·55	198·2	65·23	96·01	1·02
Average... ...	0·860	52·0	0·53	198·4	65·36	96·08	1·05

Walnut Oil (5 samples, cold pressed).

	At 15° C.					
Maximum ...	0·9260	68·0	—	—	148·43	—	5·97
Minimum ...	0·9255	67·0	—	—	147·92	—	3·17
Average... ...	0·9258	67·7	—	—	148·21	—	4·38

W. P. S.

Prussic Acid in Sweet Cassava. Carmody. (*The Lancet*, September, 1900.)—The author confirms the statement of Francis (Analyst, 1877, 4-7) that prussic acid is present in sweet cassava to a considerable extent. He finds that the prussic acid is not uniformly distributed throughout the tuber, the inner part containing on the average 0·006 per cent., whilst the skin and outer cortical layer contain 0·028 per cent. of HCN. In bitter cassava the prussic acid is uniformly distributed, the average amount being 0·023 per cent. W. P. S.

The Decomposition of Food Materials by Micro-organisms. Part I.: Fat-consuming Micro-organisms. J. König, A. Spieckermann, and W. Bremer. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 721-744, and 769-780.)—Three samples of cottonseed meal were used in the experiments described in this paper. They contained fungus spores, as well as bacteria of the hay and potato species. The fungi only increased in growth when the percentage of water in the meal was not under 14 per cent. From 14 to 30 per cent. of water was most favourable to the growth of the fungi, whilst with above 30 per cent. the bacteria obtained the upper hand. The mould flora changed with an increase of moisture. *Eurotium repens* first appeared, soon followed by *Eurotium rubrum;* with 20 per cent. of moisture the "Oïdium" species of mould-fungi were noticed, and with 25 per cent. *Penicillium glaucum.* The growth of the fungi was always accompanied with a loss of organic matter, but an increase of water. On the appearance of *Penicillium glaucum*, the fat and non-nitrogenous extractives were rapidly attacked, and also the pentosans to a small extent. A small quantity of proteid matter was converted into water-soluble organic bodies, but no ammonia was formed. The nitrogenous matter was acted on to a small degree with the liberation of nitrogen. The bacteria obtained the carbon they required from the non-nitrogenous extractives (raffinose, etc.) and pentosans, decomposing the fat only to a small extent. On the other hand, the proteids were decomposed with the production of ammonia. Experiments with pure cultures of the fungi on sterilized cottonseed meal, and also on culture media containing fat, showed the same results as above. From cultures of *Aspergillus flavus* and *Eurotium repens* on sterilized cotton meal, an enzyme was extracted by means of glycerin, which had the property of forming butyric acid from monobutyrin. This enzyme did not appear to act on cotton oil, but the fact that the amount of fatty acid increased in the experiments points to some decomposition of the higher glycerides. The fat is apparently for the most part directly converted into carbon dioxide and water by the action of the fungi. W. P. S.

Detection of Cherry Juice in Raspberry Juice and Red Wines. K. Windisch. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, iv., 817-825.)—All cherries examined by the author contained hydrocyanic acid (from amygdalin). This acid is present in the flesh of the cherries as well as in the stones, although in smaller quantity. The fermented juices of cherries, both with and without stones, always contained hydrocyanic acid. The presence of hydrocyanic acid serves as a means for detecting cherry juice in other juices. The test is applied as follows: 20 to 30 c.c. of the fruit juice are distilled until about 2 c.c. have come over. To the latter is added a drop of guaiacum tincture, together with a drop of dilute copper sulphate solution. The appearance of a blue colour indicates the presence of hydrocyanic acid. The colour is fugitive, and if very faint may be intensified by shaking out with a little chloroform, in which it dissolves. A negative test does not prove the absence of cherry syrup, as the original amount of hydrocyanic acid in the cherries may have been very small. W. P. S.

Estimation of Morphine in Opium by means of Ammoniacal Silver Chloride. **C. Reichard.** (*Chem. Zeit.*, 1901, xxv., 816.)—If the opium sample exists as an alcoholic solution, it is diluted with three times its volume of water and evaporated to its original bulk; otherwise, about 2 or 3 grammes of the material are extracted for an hour with ten to twenty times that amount of boiling water, shaking or mechanically agitating the while. The mass is filtered, and the residue washed several times with boiling water. To the filtrate and washings a small excess of ammoniacal silver chloride is added, and the whole is warmed gently. After standing some time, when the precipitate no longer increases and the liquid is clear, the deposit is collected, washed till free from dissolved silver compounds, dried, and ignited in porcelain to give a button of metallic silver. Two atoms of silver correspond with one of morphine. Experiments show that the other opium alkaloids and constituents do not reduce ammoniacal silver; but to insure absence of (adventitious) reducing matter, another portion of the sample should be extracted with water, precipitated with ammonia to throw down all the alkaloids, and the filtrate examined for any reducing power. Since the reagent contains excess of ammonia, its first effect is simply to precipitate all the alkaloids in the opium, so that the substance on the filter is a mixture of metallic silver with the other opium bases; but inasmuch as the latter are all destroyed by the ignition, this does not affect the accuracy of the process. F. H. L.

The Estimation of Mercury in Antiseptic Solutions. **G. Meillère.** (*Journ. Pharm. Chim.*, 1901, xiv., 356-359.)—When the mercury is present in the form of chloride or iodide, it can be extracted quantitatively by shaking the liquid with an excess of sulphuric ether or acetic ether. Thus, 5 c.c. of a solution of mercuric chloride containing 0·0343 gramme of mercury yielded, on evaporation of the ethereal extract, a residue containing 0·0338 gramme of mercury.

For determining the mercury in the salt thus isolated, the author precipitates the metal in the free state by means of stannous chloride or magnesium and hydrochloric acid, etc., and washes and dries the precipitate in a weighed tube, with the aid of centrifugal force.

Mercuric cyanide is usually in a sufficiently pure state, and is readily determined in its solution, but the oxycyanide, when properly prepared, is much less soluble. Hence the commercial salt is frequently a mixture in varying proportions of mercuric cyanide and oxycyanide.

In various commercial samples the author has found from 76·8 to 81 per cent. of mercury, as against 85·04 per cent. by theory.

The cyanogen can be determined by treating 0·1 gramme of the salt with standard iodine solution, in the presence of an excess of alkali-metal carbonate, and after the lapse of several minutes titrating the liquid with a standard solution of thiosulphate. C. A. M.

ORGANIC ANALYSIS.

Estimation of Nicotine in Tobacco. J. Toth. (*Chem. Zeit.*, 1901, xxv., 610.)—The sample is dried in the air or over quicklime, ground or beaten to powder, and 6 grammes of it are weighed into a 200 or 300 c.c. porcelain basin. Here it is moistened and well rubbed up with 10 c.c. of 20 per cent. sodium hydroxide, and plaster of Paris is added by degrees till the whole forms a dry powdery mass. (This is better than adding plaster to only a paste-like consistency, as recommended by Neumann.) The powder is brought into a stout 25 × 5 centimetre cylinder, where it is repeatedly agitated for an hour with 100 c.c. of a mixture of ether and petroleum spirit. Twenty-five c.c. of the clear liquid are drawn off, run into a glass basin, treated with 40 or 50 c.c. of water and 1 drop of iodeosin solution; then an excess of decinormal acid is introduced, and the solution titrated back with alkali of equal strength. The results are accurate.

Experiments on the influence of ammonia on this process show (1) that the 25 c.c. of ethereal extract may contain 0·5 milligramme of ammonia as a maximum, but that the quantity is usually less; and (2) that if the sample contained ammonium salts, 98·5 per cent. of the corresponding ammonia would be retained in the gypsum. The method proposed by Keller (ANALYST, 1898, xxiii., 235) for removing ammonia from the ethereal solution by a current of air is not trustworthy, since the loss in alkalinity of the liquid and the alkaline vapours in the escaping air are due to slight volatilization of nicotine itself. Moreover, in Keller's process nicotine is retained by the aqueous liquid when it is extracted with the mixed solvents. F. H. L.

Formaldehyde and Sulphuric Acid as a test for Alkaloids. H. Linke. (*D. Pharm. Ges. Ber.*, 1901, xi., 258; through *Chem. Zeit. Rep.*, 1901, 184.)—The above reagent gives the following colour tests with alkaloids—Colchicine: golden yellow, gradually disappearing. Hydrastinine: greenish yellow, gradually disappearing. Strychnine: green-brown on warming. Atropine: brownish, on warming "dirty greenish-grey-brown." Homatropine: brown on warming. Scopolamine: golden yellow to orange, brown-black on warming. Veratrine: yellow-brown, reddish-brown on warming. Digitaline: brick to wine-red, dark wine-red on warming. Morphine: cherry-red, then violet, grey-black on warming. This reaction is very fine and sharp. Apomorphine: violet, then rust-red and black-blue, greenish-black on warming. Codeine: violet-blue, brown-black on warming—a fine and very sharp reaction.

Cocaine, pilocarpine, cantharidine, eserine, and caffeine give no reactions either hot or cold. F. H. L.

A Reaction of Urotropine and Piperazine. Manseau. (*Bull. de la Soc. Pharm.*, Bordeaux, June, 1901; *Ann. de Chim. anal.*, 1901, vi., 339, 340.)—The author's tests are based upon the reaction used by Jorissen for the detection of formaldehyde (ANALYST, xxiii., 41).

Urotropine, or hexamethylene-tetramine, is decomposed on treatment with acids into formaldehyde and ammonia, and can thus be detected by the purple coloration changing to blue, which it then gives with morphine.

By using other opium alkaloids instead of morphine, the following colorations are obtained: codeine, blue changing to dark-green; apomorphine, violet-blue; narceïne, saffron-yellow; thebaine, greenish-yellow; and narcotine, golden-yellow.

Piperazine, or diethylene-diamine, also gives colorations when treated with opium alkaloids in the presence of sulphuric acid. Thus, with morphine and codeine there is a slight violet tint, whilst with papaverine, narceïne, narcotine, and thebaine the colours vary from bright yellow to orange and deep brown. C. A. M.

Action of Reducing Sugars on Nickel Salts in Alkaline Solution. M. Duyk. (*Bull. de l'Ass. belge.*, 1901, xv., 267, 268.)—The author's reagent consists of 25 c.c. of a 20 per cent. solution of nickel sulphate, with 25 c.c. of sodium hydroxide solution (specific gravity 1·33), 3 grammes of tartaric acid, and 50 c.c. of water.

On boiling this reagent with the smallest trace of a reducing sugar, a pronounced turbidity is produced, which eventually subsides as a black precipitate of, probably, a sub-oxide of nickel.

The advantages claimed for this reagent over Fehling's solution are that spontaneous reduction does not occur, and that the reaction is more distinct in doubtful cases. Moreover, it is not affected by normal urine, which often decolorizes Fehling's solution and its modifications.

The author has found that cobalt salts are not reduced under the same conditions, and points to the possibility of basing a method of separating nickel from cobalt on this fact. C. A. M.

Isolation of Amido-Sugars. H. Steudel. (*Zeits. physiol. Chem.*, 1901., xxxiii., 223; through *Chem. Zeit. Rep.*, 1901, 275.)—In alkaline solution phenyl isocyanate combines with glucosamine to form a substance which is very insoluble in water, and which separates from its solution in dilute acetic acid as a heavy crystalline powder having a sharp melting-point. When 2·25 grammes of glucosamine hydrochlorate were dissolved in 30 c.c. of water and 10 c.c. of normal potassium hydroxide and 1·19 grammes of phenyl isocyanate dropped in with constant agitation and cooling, the liquid quickly solidified to a thick paste. The solid matter was removed and dissolved in hot acetic acid. On cooling large rhombic crystals separated, which were recrystallized from water and dried at 110° C. On analysis they gave figures corresponding with $C_{13}H_{16}N_2O_5$. This body begins to turn brown at 200°, and melts sharply at 210° C. Isoglucosamine yields a similar product, and the author believes that other amido-sugars may be isolated by the same process. F. H. L.

Detection of Mineral Oil in Rosin Oil. D. Holde. (*Mittheil. Kgl. Techn. Versuchsanstalten zu Berlin*, 1901, xix., 39; through *Chem. Zeit. Rep.*, 1901, 232.)—Ten c.c. of the oil are dissolved in 90 c.c. of 96 per cent. alcohol at the laboratory temperature, shaking if necessary. If a large quantity of oil remains undissolved, it may be considered as a sign of the presence of much mineral oil. This may be confirmed by collecting the oil, rinsing it with a little alcohol, and determining its

refractive index. If, however, nearly all the original oil dissolves in the spirit, any undissolved matter is neglected. The whole is cautiously diluted with water till a distinct milkiness is produced, and the mixture is set aside (all night if so required). The clear alcoholic layer is poured off from the residue, which should not exceed 1 c.c., and the oil is rinsed with a few c.c. of alcohol. It is then dissolved as before in 20 c.c. of 96 per cent. alcohol, and treated with water till not exceeding 3 drops of oil separate. These are again rinsed with spirit, washed with hot absolute alcohol, dried in a basin, and examined for their refractive index. An index of below 1·5330 at a temperature between 15° and 18° C. shows the presence of mineral oil.

F. H. L.

Linseed Oil and its Adulterants. P. C. McIlhiney. (*Report to the Commissioner of Agriculture of New York State*, 1901, 1-31.)—The author states that the character of linseed oil now made in New York State does not correspond with that given in even recent text-books, for it is prepared by heating metallic oxides with a small quantity of the oil, and adding this "drier" to the main body, which is kept at a much lower temperature than in the old-fashioned method.

The figures given by the oil treated in the new way are practically identical with those for raw linseed oil.

A table is also given showing the corrections for temperature to be added to or subtracted from the readings of a glass hydrometer correct at 15·5° C. when immersed in linseed oil. According to this, the correction for 1° F. averages 0·000361, and for 1° C., 0·000650.

Hehner and Mitchell's test, based on the amount of insoluble bromine derivatives (Analyst, xxiii., 310), is regarded as a valuable one for the detection of the adulteration of linseed oil. The author has obtained results substantially in agreement with theirs.

C. A. M.

The Detection of Drying and Marine Animal Oils. G. Halphen. (*Journ. Pharm. Chim.*, 1901, xiv., 359-365 and 391-397.)—The author's test is based upon the fact recorded by Hehner and Mitchell (Analyst, xxiii., 315) that these oils (*i.e.*, the glycerides) yield insoluble derivates on treatment with bromine.

His reagent is prepared by mixing twenty-eight parts by volume of glacial acetic acid with four parts of nitro-benzene and one part of bromine; 0·5 gramme of the oil under examination is shaken in a stoppered tube, with 10 c.c. of this reagent, until the liquid is homogeneous, and is then allowed to stand.

In this way it was found that no turbidity resulted after an hour in the case of olive, almond, castor, cotton-seed, poppy, lard, and neatsfoot oil.

With the following oils no precipitate was formed, but only a slight turbidity: Jaffa sesame oil, and certain samples of the foot oil of the horse and sheep.

A marked turbidity, and eventually a heavy precipitate, were given by the following: nut oil (precipitate after five to ten minutes), hempseed, linseed, seal, cod-liver, whale, spermaceti, and Japanese fish oils.

Colza oil was distinguished by giving a turbidity which on standing separated into two layers of liquid.

The precipitates thus obtained with the different classes of oils vary in their properties. Thus, on washing the insoluble bromides of linseed oil and of fish oils with sulphuric ether to remove the excess of the reagent, and then pressing them between filter-paper and drying them in the air, heavy substances are left, which differ in their behaviour towards carbon tetrachloride.

The solutions, obtained by treating the precipitates with this solvent in the proportion of about 2·5 c.c. to each 0·1 gramme at a temperature near the boiling-point and then filtering, are very different. The linseed oil precipitate yields a perfectly clear solution, which, on cooling, deposits, little by little, a gelatinous precipitate. With fish oils, on the other hand, the solution is opalescent, and only deposits a trace of a crystalline precipitate on cooling.

The bromides of other drying oils behave like linseed in this respect, but in the case of hemp-seed oil several hours are required before the gelatinous precipitate is deposited.

The other marine animal oils mentioned above all yielded opalescent solutions in this test.

In the case of the foot oil of terrestrial animals, a clear solution, which does not yield a gelatinous precipitate, is obtained.

The author describes a series of test experiments to show that by this method it is possible to detect 10 per cent. of linseed, hempseed, whale, or fish oil in castor, cotton-seed, or olive oils. C. A. M.

Occurrence of Mixed Glycerides in Cacao Butter. J. Klimont. (*Berichte*, 1901, xxxiv., 2636.)—Cacao butter, in the author's experiments, was separated into three fractions by crystallization from acetone. The first of these melted at 64° C., had an iodine value of 0, after repeated recrystallization, and finally yielded crystals melting at 70° C. It was found to consist of a mixture of tripalmitin and tristearin.

The second fraction, consisting of granular crystals, melted between 31° and 32° C., and had an iodine value of 28·9, a saponification value of 196·4, and a composition agreeing with the formula $C_{55}H_{104}O_6$. The author concluded that it was a triglyceride containing the three radicles of palmitic, oleic, and stearic acid.

The third fraction was also regarded as a mixed glyceride. It melted at 26° to 27° C., had an iodine value of 31·7, and a saponification value of 210·5, and from an elementary analysis was concluded to have the composition $C_{51}H_{96}O_6$.

The author was unable to identify triolein in cacao butter. C. A. M.

The Use of Iodine Monobromide in the Analysis of Fats and Oils. J. Hanus. (*Zeit. für Untersuch. der Nahr. und Genussmittel*, 1901, xx., 913-920.)—For determining the iodine absorption of fats and oils the author uses iodine monobromide. This is prepared by slowly dropping 13 grammes of bromine into 20 grammes of finely powdered iodine contained in a beaker. During the addition of the bromine (which should take about ten minutes) the contents of the beaker are well stirred and kept below 8° C. Each drop of bromine causes a lump to form which must be broken up. When

all the bromine has been added, a rapid stream of carbon dioxide is passed through the beaker, to remove excess of bromine. The iodine monobromide so obtained is a dark-grey substance, having a metallic lustre. It keeps well in closed vessels, and is soluble in ethyl alcohol and glacial acetic acid. The reagent is prepared by dissolving 10 grammes of the monobromide in 500 c.c. of glacial acetic acid. The exact strength of this solution is determined by titrating 10 c.c. after the addition of 15 c.c. of 10 per cent. potassium iodide solution, with standard thiosulphate solution.

To obtain the iodine number of an oil or fat, from 0·1 to 0·7 gramme of the same is dissolved in 10 c.c. of chloroform; 25 c.c. of the above reagent are added, and the mixture is allowed to stand for fifteen minutes. Fifteen c.c. of 10 per cent. potassium iodide solution are then added, and the titration carried out as usual with thiosulphate solution. The use of starch solution at the end of the titrations may be dispensed with. With oils and fats having an iodine absorption below 100, the reaction is complete in ten minutes. The strength of the reagent should be checked in each set of determinations, as it slightly decreases. The results obtained agree well with those of the Hübl method. W. P. S.

Quantitative Separation of Cholesterin (Phytosterin) from Fats. E. Ritter. (*Chem. Zeit.*, 1901, xxv., 872.)—About 50 grammes of the fat are weighed into a 1,500 c.c. porcelain basin, using, if necessary, 100 c.c. of alcohol to rinse it out of the weighing vessel. It is then heated to the boiling-point on the water-bath, and mixed, with continual stirring, with an alcoholic solution of sodium ethoxide, made by dissolving 8 grammes of sodium (freed from petroleum and oily matter) in 160 c.c. of 99 per cent. alcohol without cooling. The whole is heated on the water-bath till the alcohol has evaporated, when 75 grammes (one and a half times the weight of the fat taken) of common salt are added together with enough water to dissolve most or all the contents of the basin. With constant stirring the liquid is evaporated to dryness, first over a naked flame, next on the water-bath, then in a drying oven at 80° C., and finally in the desiccator over sulphuric acid, beginning to reduce the mass to powder as soon as possible. The residue is extracted with ether in a large Soxhlet, employing a fat-free paper thimble with a plug of cotton-wool on top. This operation is generally finished in about nine hours. The ether in the flask is usually turbid at first owing to traces of glycerin, but these gradually adhere to the walls and remain attached, when the ether is poured off into another vessel to make room for a fresh quantity. Finally, all the ethereal extract is brought into a 750 or 1,000 c.c. flask, the solvent distilled away, the residue taken up in the minimum of alcohol, and water is added nearly to fill the flask. The precipitated substance is collected on a paper, washed with water, dried on the filter in an oven at 60° C., scraped off into a tared basin, the remainder rinsed off the paper with ether, and the whole is distilled or evaporated, dried at 100° or 120° C., and weighed. F. H. L.

Characteristics of Neroli Oil from different Sources. E. Theulier. (*Bull. Soc. Chim.*, 1901, xxv., 762-764.)—The author has examined thirty-three specimens

of Neroli oil distilled, by the method in use at Grasse, from orange-flowers collected between May 16 and June 2 of the present year.

It was found that the specific gravity varied but little whatever the origin of the oil, the extreme values being 0·869 and 0·8726, and the mean 0·8709.

The differences in the rotatory power were somewhat greater, ranging from +2° 50′ to +7° 20′ in a 100 millimetre tube at 23° C. The mean was +4° 48′.

The proportion of methyl anthranilate was determined by the method of A. Hesse and O. Zeitschel, in which this ester is precipitated in the form of a sulphate which is only soluble with difficulty in ether, but which can be dissolved in water and titrated. The average quantity in the samples of oil examined was 0·7 per cent.

The esters determined as linalyl acetate varied between 8·08 and 14·7 per cent., the mean being 11·27 per cent. This maximum was lower than in preceding years, in which the highest proportion was usually 17 to 18 per cent.

The yield of oil obtained from the flowers varied considerably with their origin. It ranged from 0·91 to 1·32 per cent. C. A. M.

Composition of Cannes Geranium Oil. Jeancard and Satie. (*Bull. Soc. Chim.*, 1901, xxv., 516-519.)—In continuation of their research on geranium oil (ANALYST, xxv., 160) the authors have made a complete examination of thirty-one samples of Cannes oil distilled at different periods between September 3 and October 6.

In the subjoined table their results are summarized, together with those of the united residual oils after neutralization, saponification, and determination of the alcohols :

Geranium Oil.	Specific Gravity at 25° C.	Rotatory Power ($l=100$) at 15° C.	Superficial Tension.	Specific Viscosity.	Saponification Value.		Solubility in 70 per cent. Alcohol at 15° C. 1 vol. in
					Hot.	Cold.	
Ordinary ...	0·8874 to 0·8906	−9°38′ to −10°55′	2·939 to 2·970	1′18″ to 1′25″	49 to 54	27 to 36	1·7 to 1·9
Neutralized	0·8777	−8°50′	2·910	1′23″	16·8	0	1·0
Acetylated ...	0·9070	−2°40′	3·042	0′39″	211·4	0	Insol. in 20
Saponified ...	0·8802	−3°0′	2·907	1′42″	0	0	1·5

From these results the authors conclude that: (1) Neutralization diminishes the specific gravity, rotatory power and surface tension, but increases the solubility; (2) acetylation increases the specific gravity and surface tension, but diminishes the specific viscosity and solubility; (3) saponification increases the specific viscosity and the solubility.

The fact that neutralization increases the solubility explains why Cannes geranium oil is somewhat more soluble than geranium oils from other sources, for it contains less free acids than the others. C. A. M.

Myrcenol. Barbier. (*Comptes Rend.*, cxxxii., 1048; through *Pharm. Journ.*, 1901, lxvii., 89.)—The alcohol obtained by the hydration of myrcene is not identical with licareol, as was stated by Power and Kleber, but is a new alcohol—myrcenol—having the constitutional formula:

$$(CH_3)_2C : CH \cdot CH_2 \cdot C(CH_3) \cdot OH \cdot CH \cdot CH_2.$$

It is a colourless, strongly-smelling liquid, boiling at 99° to 101° C. at 10-millimetre pressure, and having a specific gravity of 0·9072 at 14·5° C. The formula given above is identical with that of Tiemann for licareol (linalool), and consequently the formula of the latter needs reviewing. The acetate—a pungent, oily liquid—has an entirely different odour from that of linalyl acetate. By oxidation with chromic acid, an aldehyde, $C_{10}H_{16}O$, is obtained, which is not citral. It boils at 110° C. at 10-millimetre pressure, gives an oxime boiling at 148° to 150° C. under the same pressure, and a semi-carbazone melting at 195° to 196° C. W. P. S.

On the Bromine and Iodine Values of Proteids. W. Vaubel. (*Zeit. anal. Chem.*, 1901, xl., 470-474.)—It was shown by Blum and the author (*J. prakt. Chem.*, 1898, lvi., 393; and lvii., 365) that undecomposed albuminous bodies, such as egg-albumin and casein, were capable of absorbing the following amounts of halogens at the maximum: Iodine, 6 to 7; bromine, 4 to 5; chlorine, 2 to 3; and fluorine, about 1 per cent. It was also found that in these experiments a greater quantity of hydrochloric acid., etc., was liberated than corresponded to the degree of substitution. The author has therefore made experiments to determine the total amount of halogen used, both in the substitution and in the formation of hydrobromic or hydriodic acid by the withdrawal of hydrogen. Using a solution of bromate for the titration, and deducting 9·0 grammes of bromine for the substitution and the corresponding hydrobromic acid liberated, the following results were obtained calculated on 100 grammes of the anhydrous and ash-free material:

	Bromine.
Egg-albumin	35·04 grammes.
Blood-albumin	40·76 ,,
Casein	27·00 ,,

On dissolving these proteids in alkali, and boiling them for an equal length of time, the sulphur, split off as hydrogen sulphide and reacting with the bromine, was eliminated and less bromine absorbed. This sulphur as hydrogen sulphide was calculated from the bromine results to be as follows: Egg-albumin, 1·12; blood-albumin, 0·72; and casein, 0·45 per cent.

In determining the iodine values Dieterich's method was first used. One gramme of the air-dried albumin, etc., was treated with 50 c.c. of water in a stoppered litre-flask, and, after standing over night, mixed with 20 c.c. of $\frac{N}{10}$ iodine solution, and left for three days; 500 c.c. of water were then added, and the liquid titrated with $\frac{N}{10}$ thiosulphate solution, of which not more than 11 c.c. or less than 6·5 c.c. was required.

In this way it was found that the anhydrous and ash-free substances consumed

the following amounts of iodine: Egg-albumin, 21·345; blood-albumin, 22·285; and casein, 20·844 per cent.

Deducting $6·5 \times 2$ grammes for the iodine absorbed by substitution and the corresponding hydriodic acid liberated, the following values were obtained for the hydrogen withdrawn by the iodine without substitution:

	Iodine.	Hydrogen.		Bromine.	Hydrogen.
Egg-albumin ...	8·345	0·0657	...	35·04	0·4380
Blood-albumin ...	9·285	0·0731	...	40·76	0·5095
Casein	7·644	0·0601	...	27·00	0·3450

From a comparison with the bromination figures, these iodine values appear to be incomplete.

By treating the solution of the proteid with 10 grammes of sodium bicarbonate, and adding 100 c.c. of the iodine solution, considerably higher results were obtained, thus: Egg-albumin, 56·41; blood-albumin, 60·87; and casein, 51·88 per cent. Deducting from these values 13 grammes of iodine for the substitution process, the following figures were obtained, in which the amounts of hydrogen are in much closer agreement with the bromination results:

	Iodine. Per cent.	Hydrogen. Per cent.
Egg-albumin	43·41	0·3418
Blood-albumin	47·87	0·377
Casein	38·88	0·3061

C. A. M.

Gas-Volumetric Estimation of Chlorides and Phosphates in Urine. E. Riegler. (*Wiener med. Bl.*, 1901, xxiv., 527; through *Chem. Zeit. Rep.*, 1901, 246.)—This process depends on the liberation of nitrogen from hydrazine sulphate by means of silver chloride, according to the equation

$$4AgCl + N_2H_4.H_2SO_4 + 6NaOH = 4Ag + 4NaCl + Na_2SO_4 + 6H_2O + N_2,$$

the nitrogen being measured in any convenient instrument and converted into weight by Baumann's tables. From the chlorides of the urine the silver chloride is prepared in the usual fashion; the phosphoric acid is thrown down with magnesia mixture, the precipitate filtered and washed, and dissolved in nitric acid. The solution is mixed with silver nitrate and ammonia to a faintly alkaline reaction, and boiled for a few minutes. The silver phosphate is then decomposed with hydrochloric acid in nitric acid solution. One milligramme of nitrogen corresponds with 8·23 milligrammes of NaCl, or with 3·34 milligrammes of P_2O_5. F. H. L.

The Determination of Traces of Sugar in Urine. E. Raimann. (*Zeit. anal. Chem.*, 1901, xl., 390-402.)—In this paper the author first shows that a single precipitation of the sugar with phenylhydrazine gives unreliable results, and then describes the following modification: 500 c.c. of the urine are fermented with fresh yeast at 34° C. for twenty-four hours, and then treated at the same time as 500 c.c. of the non-fermented urine in the following manner: After the addition of 100 c.c. of a 25 per cent. solution of lead acetate, the liquid is filtered, and the excess of lead

removed from the filtrate by means of hydrogen sulphide; 450 c.c. of the second filtrate are evaporated on the water-bath to about 100 c.c., then made up to 120 c.c. and filtered. Of this filtrate 100 c.c. are heated on the water-bath for one and a half hours with 5 c.c. of glacial acetic acid and 3 c.c. of phenylhydrazine, and, after standing for twenty-four hours, the precipitate collected on a dried and weighed filter-paper, and dried first in the air and subsequently at 110° C. until constant in weight.

The difference between the weight of osazone thus obtained and that given by the unfermented urine is taken as pure phenyl-glucosazone.

In test experiments in which known quantities of sugar were added to the fermented urine the amount of osazone calculated into glucose only averaged 49 per cent. of the theoretical quantity, and in order to obtain the percentage of sugar in the urine the difference in weight of the osazone precipitates was multiplied by the factor 0·329.

In this way the following results, among others, were obtained :

Sugar Added. Per cent.	Sugar Found. Per cent.	Error. Per cent.
0·005	0·006	+ 0·001
0·100	0·095	− 0·005
0·206	0·213	+ 0·011

The fermented urine invariably yields with phenylhydrazine a non-crystalline precipitate, which adheres firmly to the bottom of the beaker. C. A. M.

The Detection of Acetanilide in Urine. A. Petermann. (*Ann. de Chim. anal.*, 1901, vi., 165.)—About 10 c.c. of the urine are boiled for a few minutes with 25 c.c. of hydrochloric acid. After cooling, 1 c.c. of a 3 per cent. aqueous solution of phenol is added, followed by 2 or 3 drops of a 10 per cent. solution of calcium chloride, the liquid being shaken after the addition of each drop. In the presence of para-amido-phenol, the decomposition-product of acetanilide, the urine acquires a red colour, which, however, it is difficult to distinguish from the coloration which normal urine gives with hydrochloric acid.

Concentrated ammonium hydroxide is next poured down the side of the glass, this reagent changing the red colour to blue. The reaction is certain in the presence of acetanilide, but may be doubtful when it is not present.

As a confirmatory test 100 or 200 c.c. of the suspected urine are mixed with a fourth of its volume of hydrochloric acid, and boiled for several minutes. When cool, the liquid is neutralized with calcium carbonate and extracted several times with ether. The latter is decanted, and shaken with water containing one-fourth of its volume of hydrochloric acid. The lower layer is then withdrawn, gently warmed to expel the last traces of ether, and tested for acetanilide as above. C. A. M.

Estimation of Ammonia in Urine. O. Folin. (*Zeits. physiol. Chem.*, 1901, xxxii., 515; through *Chem. Zeit. Rep.*, 1901, 239.)—The urine, or the solution of urea, is diluted with 400 or 500 c.c. of water, and distilled with magnesia or lime-water

for forty-five minutes (counting only the time of actual ebullition), collecting the distillate in decinormal acid. Without stopping the operation, a volume of water, roughly equal to that which has distilled over, is then added to the retort, and the process is continued for a second period of forty-five minutes, using a different receiver. On the assumption that the decomposition of the urea proceeds at a uniform speed, the amount of ammonia in the second distillate (as determined by titration), deducted from that in the first, gives the ammonia which existed as such in the urine. F. H. L.

On the Estimation of Urea in Urine. J. H. Long. (*Journ. Amer. Chem. Soc.*, xxiii., 633.)—In the opinion of the authors, the old Liebig method, in which the urea is titrated with mercuric nitrate solution, is capable of rendering good service where a number of quick comparative determinations are to be made. The method was discarded because other substances besides urea present in urine react with mercuric nitrate, and, consequently, the authors have made numerous experiments to determine the influence which these bodies exercise. The most important of these, after the chloride—the effect of which is well known and easily corrected—are ammonia, uric acid, and creatinin. The average correction for these three bodies was found to be 2·0 c.c. mercury solution, which may be used in the case of fresh urine with only a small margin of error, whilst, if the disturbing bodies themselves are estimated, the corresponding correction may be applied with considerable accuracy. A. G. L.

Estimation of Urea in Urine. O. Folin. (*Zeits. physiol. Chem.*, 1901, xxxii., 504; through *Chem. Zeit. Rep.*, 1901, 239.)—Urea is quantitatively decomposed into ammonia and carbon dioxide within half an hour when it is heated with magnesium chloride boiling in its own water of crystallization (at 160° C.). Accordingly, 3 c.c. of the sample, 20 grammes of magnesium chloride, and 2 c.c. of strong hydrochloric acid, are boiled in a 200 c.c. Erlenmeyer flask under a 200 × 10 millimetre reflux tube till the drops of condensed liquid fall with a hissing noise into the flask. The boiling is continued for another twenty-five or thirty minutes, when the whole is cautiously diluted with water, rinsed into a 1-litre flask, and distilled with 7 c.c. of 20 per cent. sodium hydroxide till about 350 c.c. have passed over. The distillate is boiled up, cooled, and titrated, every 1 c.c. of decinormal ammonia in the liquid corresponding with 3 milligrammes of urea. Corrections must be made for any ammonia in the magnesium chloride, and for pre-existing ammonia in the urine.
F. H. L.

The Analysis of Shellac. E. J. Parry. (*Chemist and Druggist*, 1901, 689.)—The writer states that samples of shellac absolutely free from rosin are the exception and not the rule. For detecting this adulteration with rosin, he relies on the determination of the acid number, the ester value, and the iodine absorption. Pure samples of shellac gave acid values between 55 and 65, with an average of 60, whilst rosins gave an average of 160. The ester values of shellacs lie between 155 and 175, those of rosins generally below 20, averaging about 10. The iodine numbers of

different samples of shellac examined lay between 4 and 10. Rosin gives an average iodine absorption of 110. W. P. S.

Detection of Chrome Yellow in Dyed Cotton. P. Cazeneuve. (*Bull. Soc. Chim.*, 1901, xxv., 761-762.)—Diphenyl carbazide is an extremely sensitive reagent for chromic acid, the limit of sensibility exceeding 1 part in 1,000,000.

The cotton fibre is treated with 1 c.c. of a 10 per cent. solution of potassium hydroxide, an immediate decolorization being effected in the presence of lead chromate. The liquid is then rendered strongly acid with acetic acid, and shaken with a pinch of diphenyl carbazide or its acetate, when a pronounced violet colour confirms the presence of chromic acid. C. A. M.

The Use of Certain Indicators by Artificial Light. A. Kufferath. (*Zeits. f. angew. Chem.*, 1901, 916.)—Experiments were carried out to ascertain whether the indicators could be relied upon for use with different sources of artificial light. The indicators used were: Methyl orange, fluorescein, cochineal, coralline, p-nitrophenol, alizarine green B, resazurine, and luteol. These were all tried by daylight, with electric glow-lamp, incandescent gas, and acetylene. From the results it appears that acetylene is the best artificial light to use where it is necessary to distinguish between two different colours, as with methyl orange, cochineal, coralline, alizarine green B, and resazurine. The sort of light is of less importance where the change is of a different nature, as with p-nitrophenol and luteol, which change from colourless to light yellow, and fluorescein, which changes from light yellow to a yellowish-green fluorescence. A. M.

The Hübl Iodine Solution. M. Kitt. (*Chem. Zeit.*, 1901, xxv., 540.)—The gradual disappearance of active iodine from the ordinary Hübl iodine solution depends on the progress of a chemical reaction which continues until equilibrium is attained. By suitable treatment of the liquid, the author finds it possible to hasten that progress, and to produce a reagent that is far more permanent in strength. Thirty grammes of iodine and 25 grammes of mercuric chloride are dissolved separately in 500 c.c. of 98 per cent. (by weight) alcohol, and the mixed solution is boiled on the water-bath under an inverted condenser for one hour. Before ebullition, the liquid contained 0·7656 gramme of iodine per 25 c.c.; after, 0·49321 gramme. In four days the boiled reagent only lost 0·75 per cent. in strength; in thirty-nine days, 8·12 per cent. Ordinary Hübl iodine loses 2·12 per cent. in one day, and 12·75 per cent. in twenty-seven days. F. H. L.

The Estimation of Hydrogen Sulphide in Coal Gas. A. Muller. (*Journ. f. Gasbel.*, xliii.; *Chem. News*, lxxxiii. (2163), 217.)—The reagents required are a solution containing 25 grammes of cadmium acetate and 200 c.c. of acetic acid per litre, and a second containing 80 grammes of copper sulphate and 175 c.c. of concentrated sulphuric acid per litre. The gas is passed at a moderate rate through 25 c.c.

of the first solution contained in an Erlenmeyer flask, the volume being accurately measured. The contents of the flask are afterwards heated to about 50° to 60° C., and the resulting precipitate (CuO) is filtered, washed with warm water, calcined and weighed. Since 1 mol. of CuO corresponds in this reaction to 1 mol. of H_2S, the volume of the latter is found by multiplying the weight of CuO by 34 and dividing the product by 79—the weight of a litre of hydrogen sulphide at the temperature of the experiment. C. S.

Determination of the Heating Power of Gases. W. Hempel. (*Zeits. f. angew. Chem.*, 1901, 713.)—The general arrangement of the apparatus is shown in Fig. 1. It consists of a burner *A*, which is so arranged that it can be fed with the gas under examination and at the same time with oxygen. It is thus possible to so

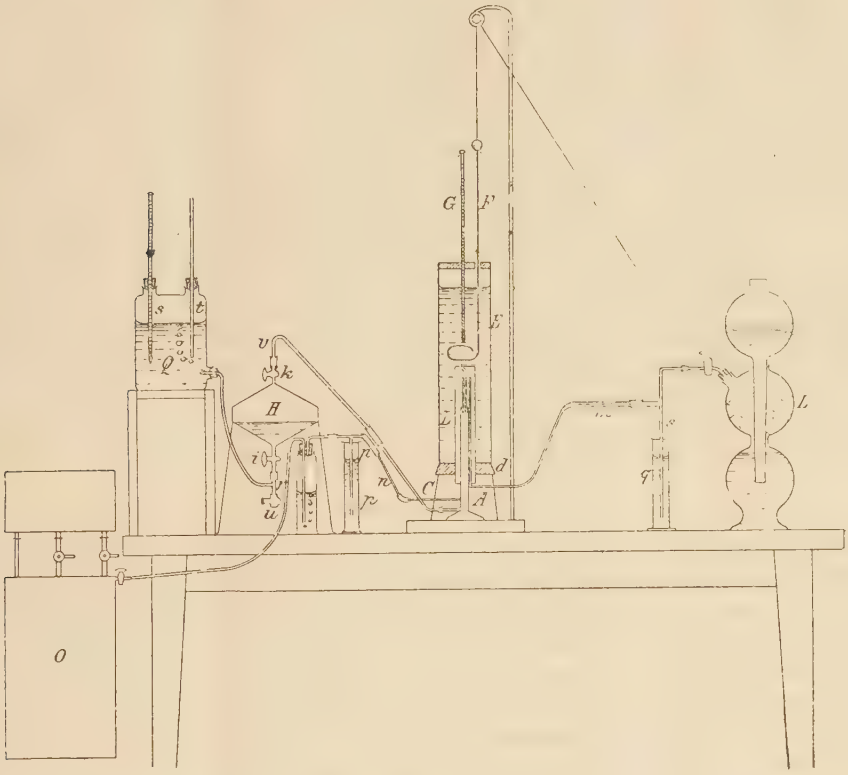


Fig. 1.

far reduce the volume of the products of combustion that they are completely cooled in a copper tube *B* only 18 centimetres long and 3·3 centimetres wide. The copper tube is closed above, and is fixed by means of the rubber cork *d* into the wide glass tube *E*, in which 500 c.c. water can be placed. The thermometer can be read to 0·02° C. The gas is contained in the reservoir *H*, which is made as flat as

possible. The gas is ignited by means of a quite small flame of hydrogen, generated in an ordinary generator *L*. The oxygen is contained in the holder *O*.

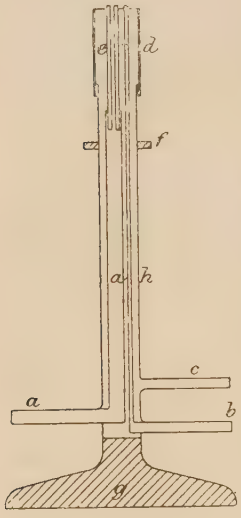


Fig. 2

In order to render it possible to supply exactly the same quantities of hydrogen and oxygen in different determinations, the gases are led through the capillary tubes *m* and *n*, and their flow is regulated so that no bubbles rise out of the cylinders *p* and *q*.

The construction of the burner is shown in Fig. 2. The gas under examination enters by the tube *a*, hydrogen by the tube *b*, and oxygen by *c*. The tubes are all provided with porcelain tips, *d*, *e*, to prevent conduction of heat. The gas reservoir *H* is connected with the Woulffe bottle *Q*, so arranged that the pressure of the gas remains practically the same throughout. The end of the combustion is indicated by the cessation of the bubbles from *t*. The apparatus is calibrated by burning a reservoir full of pure hydrogen. The small ignition flame is kept burning exactly the same time in each determination; thirty seconds should be long enough. The heating effect of this is determined separately.

For roughly comparative purposes, the relative heating power of gases may be ascertained by observing the length of flame they give under identical conditions. For this purpose a one-hole burner is provided with a glass chimney on which graduations are marked. The pressure and temperature of the gases must, of course, be in each case the same. The results are somewhat affected by the specific gravity of the gas, for light gases under the same pressure pass out more rapidly than heavy ones. A. M.

INORGANIC ANALYSIS.

The Determination of Platinum and Iridium in Platinum Ores. Leidié and Quennessen. (*Journ. Pharm. Chim.*, 1901, xiv., 351-355.)—The following modification of Leidié's method (ANALYST, xxvi., 108) is recommended when only the amount of platinum and available iridium in an ore are required: Five grammes of the ore are repeatedly extracted with hot *aqua regia* (nitric acid, specific gravity 1·32, 1 part; hydrochloric acid, specific gravity 1·18, 3 parts) until nothing more dissolves. The extract is evaporated to dryness at 105° to 110° C., and the residue dissolved in a small quantity of water and filtered.

The filtrate is then brought to about 70° C., and treated with sodium nitrite until the liquid becomes neutral to turmeric, when sodium carbonate is added little by little until no more precipitate is formed. The liquid is then filtered, after being heated to boiling, in order to remove the foreign metals.

Sodium hydroxide is now added to the filtrate until the reaction is alkaline, and the osmium and ruthenium expelled as volatile peroxides by means of a current of chlorine, which is passed through the liquid maintained at 70° to 80° C

The liquid is next neutralized with hydrochloric acid, and any chlorides that may have been formed reconverted into nitrites by the addition of more sodium nitrite. The solution will now only contain alkali salts and the double nitrites of platinum, palladium, iridium, and rhodium.

Estimation of the Platinum.—At this stage in the original method the liquid was saturated with ammonium chloride, but, instead of this, from 30 to 35 per cent. of potassium chloride are introduced. The double nitrites of rhodium and iridium with potassium, which are precipitated, are insoluble in solutions of alkali chlorides.

The nitrites of platinum and palladium in the filtrate are converted into chlorides with hydrochloric acid, and the mass dissolved in boiling water. A slight excess of formaldehyde is now added, and the metals precipitated on boiling the liquid are ignited, reduced in a current of hydrogen, and dissolved in *aqua regia*. The residue left on evaporation of this solution is dissolved in water and treated with a reducing agent, to convert palladic chloride into palladous chloride, after which the platinum can be precipitated with ammonium chloride, and estimated in the usual way.

Estimation of the Iridium.—In a portion of liquid from which the osmium and ruthenium have been expelled, the double nitrites of rhodium and iridium are converted into chlorides by means of *aqua regia* containing but little nitric acid.

The iridium is then precipitated by means of ammonium chloride, and the precipitate washed with ammonium chloride solution, ignited, reduced in hydrogen to the metallic form, and weighed.

The iridium could also be precipitated by means of potassium chloride as the double chloride $2KCl.IrCl_4$. The potassium chloride is removed by washing with water, and the residue reduced, dried at 115° C., and weighed.

The authors point out that this method only gives the available iridium, since any which is present in the free state or in combination with the osmium is insoluble in *aqua regia*, and is left in the residue in the preliminary extraction. C. A. M.

The Estimation of Tin in White-metal Alloys. Frederick Ibbotson and Harry Brearley. (*Chem. News*, 84, 167.)—Tin may be readily estimated by reducing the hot solution of the chloride, cooling in an atmosphere of CO_2, and titrating with iodine and starch. The reduction can be conveniently effected by means of iron, but any excess added must be dissolved completely. If antimony is present, it will be precipitated as metal, and cannot be dissolved again, but as cold acid solutions of stannic chloride are not reduced by antimony, which readily reduces them on heating, the solution may be directly titrated with iodine as usual, very good results being obtained. The antimony may be filtered off after the titration and estimated, but the results obtained are rather low.

An improvement on the above method consists in reducing the stannic chloride with finely-powdered metallic antimony. The reduction of 0·15 gramme of tin is complete after one minute's boiling, and the excess of antimony which remains undissolved acts as a safeguard during the cooling, since it reduces any tin which may have become oxidized whilst the solution is still hot. Cold solutions of stannous

chloride take up oxygen less readily. The test analyses given show that the reduction is complete.

The influence of various substances likely to be present in an ordinary analysis on the above method was examined, and it was found that the presence of iron, chromium, nickel, zinc, manganese, aluminium, bismuth, phosphorus, and sulphur is without effect on the results. The quantity of hydrochloric acid present should always be about one-fifth of the total volume. If copper is present, it will be reduced to the cuprous state, but accurate results may nevertheless be obtained if the iodine is added drop by drop to the vigorously agitated solution, so as to prevent the formation of a local excess of iodine. It is also advisable to have rather more hydrochloric acid present, up to about one-third of the total volume. Cobalt apparently gives very slightly high values. Lead is without influence if sufficient hydrochloric acid is present to prevent the formation of lead iodide. The presence of arsenic completely vitiates the results, whether the tin is reduced with iron or with antimony. Mercury is reduced to the metallic state, but is not oxidized in cold solutions. If molybdenum or tungsten is present, a coloured lower oxide is formed, but this is not appreciably reoxidized by the iodine, and the starch blue can be readily distinguished. A. G. L.

The Determination of Sulphur in Iron and Steel. William A. Noyes and L. Leslie Helmer. (*Journ. Amer. Chem. Soc.*, xxiii., 675.)—The authors recommend the following two methods as being more rapid and accurate than those now in use: In the first, 5 grammes of the steel are added in portions at a time to 200 c.c. of water and 8 c.c. of bromine contained in a flask, cooling after each addition. The solution is finally boiled for a moment to expel excess of bromine, filtered, the filtrate added to 130 c.c. of 10 per cent. ammonia contained in a 500 c.c. flask, the liquid made up to the mark, and well mixed. Three hundred c.c. are then filtered off, evaporated in a wide beaker to 100 c.c., and precipitated with 10 c.c. of barium chloride solution, after the addition of 1 drop of hydrochloric acid of specific gravity 1·12. After the precipitate has settled, it is filtered off, ignited, and weighed as usual. The filter-paper containing the original insoluble residue is burnt with some sodium carbonate, the oxidation being completed by adding a little potassium nitrate. The melt is dissolved in water, the solution filtered, acidified with hydrochloric acid, and precipitated with 5 c.c. of barium chloride solution. Three-fifths of the weight of barium sulphate found here is added to the main quantity of barium sulphate found, and the total represents the sulphur in 3 grammes of iron.

In the second method, 5 grammes of the iron, mixed with 7 grammes potassium chlorate, are added in portions at a time to 120 c.c. nitric acid (specific gravity 1·20) and 1 gramme potassium bromide, cooling if necessary. When solution is complete, the liquid is evaporated to dryness in a dish, the residue dissolved in hydrochloric acid and filtered after diluting, both filtrate and residue being treated as in the first method. In the bromine method, the amount of sulphur left in the insoluble residue is larger than in the nitric acid method, but no evaporation to dryness is required, and practically no fumes are given off during solution.

Test experiments, in which known amounts of ammonium ferrous sulphate were

added to sulphur-free iron, and to steel containing known amounts of sulphur, gave satisfactory results. The methods also gave concordant and higher results than Blair's aqua regia method, but the values obtained by using the latter are rather irregular. Since the quantities of sulphur left in the residues will probably prove nearly constant for any given class of steel, the authors believe that it will be possible to add an empirical correction to the quantity found in the solution, thus shortening the process very considerably. A. G. L.

Ammonium Persulphate as a Substitute for Lead Peroxide in the Colorimetric Estimation of Manganese. Harry E. Walters. (*Proc. Engineers' Soc., Western Pennsylvania*, xvii., 257.)—In the following method the manganese is oxidized by means of ammonium persulphate in the presence of a silver salt: Samples of 0·2 gramme each of the test-steel and of a steel of known percentage of manganese are placed in test-tubes, and 10 c.c. of nitric acid (specific gravity 1·20) added to each. The tubes are heated in a water-bath till solution is complete and nitrous fumes are driven off. Fifteen c.c. of a solution of silver nitrate containing 0·02 gramme of $AgNO_3$ are added, and then immediately 1 gramme of solid ammonium persulphate, and the tubes again warmed, when oxidation will commence. After another half-minute's heating the tubes are placed in a cold-water bath. After oxidation is over and the tubes are cool, they are compared as usual.

Pig-irons and slags are treated in the same way as steels, except that slags should be moistened before adding the acid, and the solution filtered from suspended matter. In this case, the 15 c.c. of silver nitrate solution are used to wash the filter-paper. In dissolving shot or chilled iron samples, a little persulphate may be added to aid the solution of the sample.

The advantages claimed for the method are that it does away with the decantation of the solution from the excess of lead dioxide, that less acid is used, and that the oxidation is effected more rapidly at a lower temperature. The results quoted show that the two methods furnish practically identical results.

In experimenting with the method, it was found that solutions of ammonium persulphate could not be used instead of the solid, and also that if the salt was perfectly dry the reaction was incomplete. The author makes it a practice to add 10 c.c. water to each 1 lb. bottle of the dry salt a day or two before use.

A. G. L.

Detection of Small Quantities of Nickel in the Presence of Cobalt. H. Ditz. (*Zeits. f. angew. Chem.*, 1901, 894.)—Advantage is taken of the property of tartaric acid to prevent the precipitation of cobalt; but, instead of precipitating with hydrogen sulphide, as in Villiers' process (Analyst, 1900, 305), the nickel is precipitated as chromate.

The neutral solution is placed in a capacious flask, and potassium chromate is added in slight excess. It is heated nearly to boiling, and 5 to 10 grammes of Rochelle salt are added. After this is all dissolved, the liquid is boiled vigorously for several minutes; then it is allowed to cool and diluted with water, if it be

coloured very intensely. Any cobalt chromate precipitated is thus brought into solution again; but if nickel be present, a brown precipitate settles to the bottom of the flask, where it can be readily detected in the green liquid, even though the amount of nickel be very small. The precipitate contains a small proportion of cobalt. A. M.

The Quantitative Separation and Determination of Uranium. Edward F. Kern. (*Journ. Amer. Chem. Soc.*, xxiii., 685.) As the result of his investigation, the author finds that the separation of uranium from iron, nickel, cobalt, zinc, and manganese is best effected by means of either a saturated solution of sodium carbonate, in which the uranium is completely soluble on prolonged boiling, or else by extracting the iron with ether and separating uranium from the other metals by means of a saturated solution of ammonium carbonate. In this method, which is to be preferred to the first, the solution of the mixed chlorides in hydrochloric acid (of specific gravity 1·10) is extracted three times with ether free from alcohol, which has previously been shaken with hydrochloric acid of the same density. From the alkalies and alkaline earths, uranium may be separated by electrolysis in the presence of sodium acetate and acetic acid; by repeated precipitation with ammonia in hot solutions containing ammonium chloride; or by precipitation with ammonium phosphate in the presence of ammonium acetate, the precipitate in this case being boiled for at least fifteen minutes before filtering. The pyrophosphate should be heated to low redness in a porcelain crucible; should reduction occur, it may be remedied by moistening with strong nitric acid and reigniting.

The author also finds that the reduction of U_3O_8 to UO_2 is unreliable as a check, and that the most rapid method of determining uranium consists in reducing the sulphate solution to $U(SO_4)_2$ with zinc, aluminium, or magnesium, and titrating with potassium permanganate. In hydrochloric acid solution, or with stannous chloride as a reducing agent, the reduction is carried beyond UCl_4, sometimes reaching UCl_3, and the results are consequently utterly unreliable. The preceding methods were tried practically in estimating uranium in pitchblende, those based on the ether separation giving the best results. A. G. L.

The Precipitation and Separation of Silver Electrolytically. W. H. Fulweiler and Edgar F. Smith. (*Journ. Amer Chem Soc.*, xxiii., 582.)—A series of experiments was made in which silver was deposited electrolytically from cyanide solutions (2 to 5 grammes KCN per 125 c.c.) containing, in addition to the silver, copper, copper and cadmium, copper, cadmium and zinc, and copper, cadmium, zinc, and nickel. The time required to deposit 0·1 gramme of silver at a temperature of 55° to 80° C. was three to six hours, using a current of about 0·02 ampères for a surface of 100 square centimetres, the voltage being 1·2. The results obtained were satisfactory; but if both cadmium and zinc are present, it appears to be essential to heat the liquid to 75° or 80° C. before passing the current to prevent the precipitation of some of the cadmium, and the presence of nickel slightly retards the deposition of the silver. A. G. L.

The Electrolytic Separation of Mercury from Copper. C. Roscoe Spare and Edgar F. Smith. (*Journ. Amer. Chem. Soc.*, xxiii., 579.)—In reply to some criticisms on this method the authors give results of a series of experiments in which mercury was separated from copper electrolytically in potassium cyanide solution (2 to 6 grammes KCN per 125 c.c.), the quantity of copper present being from one-third to eleven times the weight of the mercury. The current used was 0·1 to 0·6 ampères for a surface of 125 square centimetres, the voltage being 1·1 to 1·7, the temperature of the liquid 60° to 65° C., and the time required to deposit 0·12 gramme of mercury, two to five hours. All the results obtained were satisfactory, and the deposit was always perfectly free from copper. Some experiments in which mercury was deposited from solutions containing copper and cadmium, and copper, cadmium and zinc, also gave good results. A. G. L.

The Electrolytic Method applied to Uranium. L. Gavit Kollock and Edgar F. Smith. (*Journ. Amer. Chem. Soc.*, xxiii., 607.)—The authors obtained good results by electrolyzing solutions of uranium acetate containing a little free acetic acid, as well as solutions of uranium sulphate and nitrate. The uranium is deposited as hydrated protosesquioxide, ignited and weighed as U_3O_8. The dilution used was 125 c.c. for 0·1 to 0·2 gramme U_3O_8, the temperature 65° to 75°, and the duration of each experiment four to seven hours. The current employed for 107 square centimetres of cathode surface was 0·05 to 0·55 ampères and 4 to 16 volts in the case of the acetate solutions, and 0·02 to 0·04 ampères and 2 to 4 volts for the sulphate and nitrate solutions. Good results were also obtained in separating uranium from barium, calcium, magnesium, and zinc in solutions of the acetates, provided that evaporation of the liquid was prevented as far as possible, as otherwise the uranium hydrate tends to enclose the separated salts, which cannot then be washed out. This method, however, cannot be used to separate uranium from iron, nickel, or cobalt. A. G. L.

The Electrolytic Determination of Molybdenum. L. Gavit Kollock and Edgar F. Smith. (*Journ. Amer. Chem. Soc.*, xxiii., 669.)—The authors found that no deposition took place on passing a current through neutral solutions of sodium molybdate heated to 75° C. On adding 2 drops of concentrated sulphuric acid, however, the liquid turned dark blue, and then, as this colour gradually disappeared, the cathode became coated with a black deposit of the hydrated sesquioxide. Concordant results could not be obtained by merely washing and drying this deposit, but very satisfactory results were found by dissolving the deposit in dilute nitric acid, evaporating the solution to dryness, and heating the residue of molybdic acid. The electrolyte contained 0·1 to 0·25 gramme MoO_3 in 125 c.c.; the current used was 0·02 to 0·04 ampère and 2 volts for a cathode surface of 107 square centimetres; and the time required for the deposition was two to four hours.

It was also found that, instead of acidulating the solutions with sulphuric acid, acetic acid (1 c.c. of 29 per cent.) could be used; the time required in this case is rather longer, and it is best to add the acetic acid gradually. Since the presence

of sodium acetate was found not to interfere with the electrolysis, the method was applied to the analysis of molybdenite as follows: The mineral was fused with sodium carbonate and nitrate, the melt dissolved in water, the solution filtered from insoluble oxides, and the filtrate electrolyzed after acidulating with acetic acid and boiling off the carbonic acid. In the liquid left after the electrolysis, the sulphur was estimated as barium sulphate. The results obtained were fairly satisfactory.

A. G. L.

On the Solubility of certain Metallic Oxides in Solutions of Sodium or Ammonium Salicylate. J. Wolff. (*Zeit. anal. Chem.*, 1901, xl., 459-462.)—Concentrated solutions of sodium or ammonium salicylates possess the power of dissolving freshly-precipitated metallic oxides, the amount dissolved depending on the nature of the oxide. Copper hydroxide is exceedingly soluble even in the cold. Iron hydroxide and aluminium hydroxide, however, do not dissolve until the temperature reaches 80° C., and are both much less soluble than copper hydroxide.

On adding a slight excess of sodium hydroxide to the solutions in sodium salicylate, the red iron solution becomes lighter in colour, but there is no change in the copper or aluminium solutions. In the presence of a large excess of sodium hydroxide the iron solution gives a precipitate of ferric hydroxide, the copper solution changes from emerald green to bright blue.

A slight excess of ammonium hydroxide does not change the appearance of any of the ammonium salicylate solutions, whilst a large excess of ammonia produces a blue coloration in the copper solution, but does not give any precipitate with either the iron or aluminium solutions.

Hydrogen sulphide completely precipitated the iron as sulphide.

The solubility of copper hydroxide in concentrated sodium salicylate solution depends upon the formation of copper-sodium salicylate, which the author has obtained in a crystalline condition. A solution of this salt is reducible by glucose.

The author explains the formation of this compound by the following equation:

$$2C_6H_4\begin{matrix}COONa\\OH\end{matrix} + Cu\begin{matrix}OH\\OH\end{matrix} = \begin{matrix}C_6H_4-COONa\\O\\|\\Cu\\|\\O\\C_6H_4-COONa.\end{matrix} + 2H_2O.$$

C. A. M.

Estimation of Chlorates in Electrolytic Bleach and Chlorate Liquors. H. Ditz. (*Chem. Zeit.*, 1901, xxv., 727.)—The process which the author has already described (Analyst, 1900, xxv., 80) for the iodometric analysis of mixtures of chlorates and hypochlorites has been reinvestigated and modified slightly to suit the purposes indicated in the title. The apparatus remains as before, but the main bottle should hold 1·5 litres, and the washing flask, which is now a cylinder, about

20 c.c. It has been found that the one hour's standing is not necessary, the reaction being finished well within five minutes. The volume of strong hydrochloric acid must not be reduced below 50 c.c., and 500 or 600 c.c. of diluting water should be employed in order to hinder any action between that acid and the potassium iodide. The method now runs as follows: If a chlorate liquor is to be analysed, a volume containing about 0·1 gramme of $KClO_3$ should be taken; if a determination of chlorate in mixtures of chlorates and hypochlorites, the volume should be such that between 40 and 50 c.c. of decinormal thiosulphate shall be required in the final titration. If, in the latter case, 25 c.c. or less of the liquor are sufficient, 50 c.c. of strong hydrochloric acid are employed; if the liquor is weaker and more must be taken (*e.g.*, 50 c.c.), 100 c.c. of acid and a correspondingly larger quantity of diluting water are used. The liquor is brought into the flask with 10 c.c. of 10 per cent. potassium bromide solution, and after the washing tube has been two-thirds filled with 5 per cent. potassium iodide, the 50 c.c. of strong HCl are run in. The stopper of the washing tube is immediately inserted, and the apparatus allowed to rest for five minutes. Then 500 or 600 c.c. of water are introduced and 20 c.c. of 5 per cent. potassium iodide. After thorough shaking, the contents of the side tube are blown and rinsed back into the flask, and the liquid is titrated with thiosulphate. Hypochlorites must be determined separately, and their proportion deducted. Commercial chlorate liquors can be run hot into the flask as soon as the free chlorine has been boiled off, and the whole process can be finished in half an hour. It is quicker than, and quite as accurate as, the ferrous sulphate method, which latter cannot be used if much hypochlorite is present, and in which the reagent needs standardizing every day.

F. H. L.

Estimation of Phosphoric Acid in Soil by the Götz Process. E. Gully. (*Chem. Zeit.*, 1901, xxv., 419.)—By making certain modifications in this process, the author has rendered it simpler in operation, capable of dealing with larger quantities of phosphoric acid, and more suitable for the analysis of soils. The vessel in which the mechanical agitation is conducted is about 17 centimetres long, holding 65 to 70 c.c. The narrow part holds altogether 0·4 c.c., is 40 to 45 millimetres long, and is graduated into eighty equal divisions, two of which contain 5 cubic millimetres, and hold ammonium phospho-molybdate equivalent to about 0·23 milligramme of P_2O_5. The whole graduated tube thus holds a precipitate equal to 18·45 milligrammes of P_2O_5; but as Von Jüptner has already shown in the case of iron analysis, the yellow precipitate does not consolidate itself evenly in the tube, which must be calibrated with mercury, and checked on a large number of blank experiments with pure disodium phosphate. (Gully appends to his paper a table of the readings of a tube in which 80 divisions are equal to 18·43 milligrammes of P_2O_5, whereas 10, 20, and 40 are equal to 2·22, 4·42, and 9·10 milligrammes respectively.) In carrying out an analysis of soil, a quantity of the sample (from 2 to 10 grammes), which contains less P_2O_5 than the maximum capacity of the tube, is brought into acid solution, and the silica removed. Hydrochloric acid is driven off by repeated evaporations with strong nitric acid, the dry residue is taken up in 1·19 nitric acid, and the solution is diluted to 50 c.c. with the same liquid. The graduated portion

of the agitation tube is filled with 25 per cent. ammonium nitrate solution, carefully avoiding air bubbles, 25 c.c. of the soil solution are pipetted in, another 10 c.c. of ammonium nitrate are introduced, and the whole is placed for at least ten minutes in a water-bath at 65° C. It is next taken out, 25 c.c. of Finkener's* molybdate solution are added, and thoroughly agitated (by hand) for one minute, watching that the liquid in the graduated portion of the tube mixes with the rest. The tube is then filled up with the ammonium nitrate solution, and put back in the water-bath, where it should remain immersed up to its neck for twenty minutes. It is next taken out, adjusted in the centrifugal machine, and revolved for four minutes at a speed of 1,100 to 1,200 revolutions per minute. The volume occupied by the precipitate is finally read off and calculated into P_2O_5.

If it is preferred to use less than 25 c.c. of the soil solution for precipitation, that quantity of fluid must be made up by adding the requisite amount of nitric acid. It is necessary that the nitric acid should have the specific gravity specified, and that the prescribed volume of it should be present; in all other respects, notably the temperature of the water-bath, adherence to the details given above is essential. To show the accuracy of the process, Gully gives a table of sixty-five moorland soils containing from 0·03 to 0·33 per cent. of P_2O_5 when analysed by the ordinary gravimetric method, the measurement figures for which show maximum differences of +0·009 and −0·01 per cent.; and he points out that, when the discrepancy is larger, the fault is probably due to the gravimetric process, which contains greater possibilities of error than the modified Götz method. F. H. L.

Determination of Phosphoric Acid as Phospho-Molybdic Anhydride. A. Seyda. (*Chem. Zeit.*, 1901, xxv., 759-768.)—The investigations described with great minuteness in this lengthy article fall into three divisions, including attempts to simplify the Meineke-Woy process (ANALYST, 1897, xxii., 250 and 333) for determining phosphoric acid with molybdenum only, a study of the effect of adding citric acid to the molybdate reagent in order to obtain a precipitate free from uncombined molybdic acid, and the elaboration of a method of converting the Wagner-Stutzer combined molybdenum-magnesium process into one in which the magnesian precipitation is avoided. The results of the various experiments prove: (1) In the determination of phosphoric acid as ammonium phospho-molybdate, the only source of error is a contamination of the precipitate with free molybdic acid—Woy's idea that "alkali" may be present being quite incorrect. (2) Using excess of molybdate solution contamination of the first precipitate with molybdic acid cannot be avoided with absolute certainty—whence it follows that no mere agitation process can be considered universally applicable. (3) If the phosphoric acid is only precipitated once, such contamination is most likely to be prevented by mechanically agitating the mixed liquids for fifteen minutes at the atmospheric temperature, while an addition of 20 c.c. of 10 per cent. citric acid solution to the whole is advisable. In this case, if iron is present, a temperature of 20° C. must not be exceeded, and the liquid must be filtered

* One hundred and sixty grammes of ammonium molybdate dissolved in 795 c.c. of cold water, and 320 c.c. of 0·925 ammonia, poured into a mixture of 1,710 c.c. of 1·2 nitric acid with 1,205 c.c. of water, preventing any rise of temperature by constant cooling.

fifteen minutes after the end of the agitation. In the absence of iron the temperature may be allowed to rise to 30° C. (4) The precipitate is best purified from molybdic acid by dissolving it in ammonia, adding 50 to 100 c.c. of "dilute molybdate" (see below) and reprecipitating hot with nitric acid. In certain circumstances the process of purification must be repeated several times; if so, the strengths and volumes of the reagents employed must be kept as specified. (5) The precipitate can be filtered hot, and washed with "washing liquid" at 60° or 80° C. The filtrate and washings should be preserved for twenty-four hours to note any imperfect precipitation of phosphoric acid. (6) The precipitate should be rinsed down from the sides of the Gooch crucible as it creeps up with 95 per cent. alcohol. (7) The yellow precipitate is only to be considered as completely converted into phospho-molybdic anhydride when it exhibits a uniform black and crystalline appearance all through.

The reagents required for the "Wagner-Meineke-Woy" process are as follows: (1*a*) *Wagner-Stutzer Molybdate:* 150 grammes of ammonium molybdate are dissolved in about 600 c.c. of warm water in a 2-litre flask; when cool, 1 litre of 1·19 nitric acid is introduced, and to the clear mixture 400 grammes of solid ammonium nitrate are added in small portions at a time, shaking repeatedly. The liquid is diluted to the mark, kept for several days and then filtered. So prepared, separation of molybdic acid will not occur. (1*b*) *Citric Acid Molybdate:* 10 grammes of powdered citric acid are dissolved in 1 litre of the above and filtered after twenty-four hours. This reagent is much more permanent than the foregoing. (2) *Nitric Acid:* 25 per cent., specific gravity 1·15; stored in a bottle with a rubber cork bearing a 20 c.c. pipette. (3) *Ammonia:* 8 per cent., specific gravity 0·967; stored in a bottle with a 10 c.c. pipette. (4) *Dilute Molybdate:* a solution containing 0·1 per cent. of ammonium molybdate and 10 per cent. of ammonium nitrate, in a wash-bottle. (5) *Washing Liquid:* 5 per cent. ammonium nitrate and 1 per cent. nitric acid. (6) *Alcohol:* 95 per cent. in a wash-bottle.

The original solution in which phosphoric acid has to be determined is precipitated with the molybdate according to Wagner's directions, placing the beaker for twenty or thirty minutes on the water-bath and then letting it settle. [It would appear that 60° C. is the "critical temperature" for the deposition of molybdic acid, and the liquid should be kept below that point. The rule applies even when citric acid is present, as that reagent loses its inhibitory power at the same temperature.] When clear, the liquid can be either filtered at once, or safely left for hours, even overnight. The precipitate is collected on a paper, well washed with the washing liquid, and the bulk of it is rinsed into a wide flat beaker with the dilute molybdate solution, which is ready in a wash-bottle. The residue on the paper is then dissolved by allowing 10 c.c. of the ammonia to run over its surface, but the paper is preferably not perforated. Finally the filter is washed three times with cold dilute molybdate. The ammoniacal solution should be quite clear, and always is so, as if the molybdate reagent is properly prepared iron is never thrown down with the yellow precipitate. The beaker is covered with a glass, placed on gauze, heated till the liquid begins to boil, removed from the flame, and 20 c.c. of the nitric acid are added boiling hot. After two or three gentle agitations the vessel is stood on the water-bath till the solution is clear, and then set aside. It is ready for filtration in five or ten minutes.

Filtration is done in a Gooch crucible, the precipitate being collected and washed with hot washing liquid. Ignition is performed in a modified form of the apparatus described by Woy. F. H. L.

The Examination of Enamel and the Fusibility of Silicates. E. Kochs and F. Seyfert. (*Zeits. f. angew. Chem.*, 1901, 719.)—The authors give a rule for calculating from their composition a fusibility coefficient for the silicates used for enamelling iron. The ratio of the number of molecules of alumina to the number of molecules of flux is divided by the ratio of the number of molecules of silica to the number of molecules of alumina. Or, expressing the rule algebraically:

$$q = \frac{T^2}{173{\cdot}4} F K.$$

where q = function of fusibility.

T = per cent. Al_2O_3.

K = ,, ,, SiO_2.

F = sum of the figures obtained by dividing the percentage of the other (flux) constituents each by its molecular weight.

The higher the function q is, the higher is the melting-point of the enamel. With the aid of this formula it is possible to calculate the addition or additions which must be made to raise or lower the function to any required extent. Examples are given of a number of actual determinations and experiments. A. M.

The Behaviour of Borax when Distilled with Methyl Alcohol. E. Polenske. (*Arb. Kaiserl. Gesundheit*, 1900, xvii., 564-568; through *Zeit. für Untersuchung. der Nahr. und Genussmittel*, 1901, iv., 801.)—When borax is distilled with methyl alcohol, 57 to 59 per cent. of the boric acid contained in the borax passes over in the distillate; about 50 per cent. comes over readily, the remainder slowly. From the methyl alcohol remaining in the distillation-flask, sodium metaborate, $NaBO_2 + 5CH_3OH$, crystallizes out. It was found that on fusion 1 molecule of borax would drive out the carbon dioxide from 3 molecules of sodium carbonate. The melt obtained by fusing together 1 molecule of borax and 1 molecule of sodium carbonate, and which had the composition of sodium metaborate, when distilled with methyl alcohol, gave a considerable quantity of boric acid in the distillate. Distillates from the melts containing more sodium carbonate were free from boric acid. When the different melts were dissolved in methyl alcohol, the substance $NaBO_2 + 5CH_3OH$ always separated out. Sodium metaborate, on distillation with methyl alcohol, gives off boric acid until the residue has the composition $Na_{10}B_8O_{17} (= 5Na_2O + 4B_2O_3)$. This residue must be considered as a mixture of sodium metaborate and soda. The latter prevents the formation of boric esters and the possibility of distillation. W. P. S.

Determination of Boric Acid. H. Lührig. (*Pharm. Centralh.*, 1901, xlii., 50-56; through *Zeit. für Untersuch. Nahr. und Genussmittel*, 1901, iv., 801, 802.)—

The author confirms the accuracy of Jörgensen's method for the estimation of boric acid (see also the ANALYST, 1900, xxv., 101, 102). By combining Gladding's method with that of Jörgensen's, less accurate results were obtained than with the latter alone. W. P. S.

The Quantitative Determination of Ozone. A. Ladenburg and R. Quasig. (*Ber. deutsch. chem. Ges.*, xxxiv., 7, 1184.)—It was recently pointed out by Ladenburg (*Berichte*, xxxiv., 631) that the present methods for the estimation of ozone suffer from the want of a check, and are consequently unreliable. It was also shown (*loc. cit.*) that by direct weighing of the ozone, even when mixed with oxygen, such a check could readily be obtained. Evidently this method itself might be used for the estimation of ozone, but it is not generally applicable, as it demands certain conditions which cannot always be fulfilled in practice. By its help, however, it is now possible to determine the reliability of the various titrimetric methods in common use, and the examination of the potassium iodide method forms the subject of the present paper. The authors reserve to themselves the right to examine other methods in the same way later on.

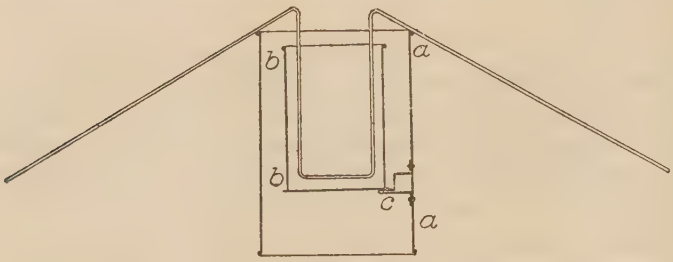
In the potassium iodide method it is usual to lead the gas through the aqueous solution of the salt acidified with an equivalent amount of sulphuric or acetic acid, and titrate the liberated iodine with sodium thiosulphate solution. Proceeding in this way, the authors always obtained results about 50 per cent. too high, and consequently adopted the method in which the ozone is led through a neutral solution, which is only acidified just before the titration. This method gives very accurate results and has the advantage that there is no return of the blue colour, which always occurs in the first method after the lapse of several hours, and necessitates the addition of a further considerable amount of sodium thiosulphate. This result is quite contrary to that found by Brunck (*Berichte*, xxxiii., 1835) and to the current opinion. The high results obtained by the first method may be due to a catalytic action of the ozone on the oxygen and the solution of potassium iodide, or else to the formation of hydrogen peroxide during the reaction according to the equation—

$$4O_3 + 10HI + H_2O = 5I_2 + H_2O_2 + 5H_2O + 3O_2,$$

which represents the quantities found almost quantitatively.

The actual determinations were carried out as follows: Dry oxygen was passed through a glass bulb of a capacity of nearly $\frac{1}{2}$ litre, provided with two stopcocks, until the weight of the closed bulb remained constant. Ozone was then passed through for about twenty minutes, and the bulb again weighed. The difference between the two weighings, multiplied by 3, gives the weight of ozone present. The bulb was then connected to two wash-bottles containing potassium iodide solution, and the ozone slowly sucked through them by means of a pump. When a neutral solution of potassium iodide was employed, only a trace of iodine was found in the second wash-bottle. An equivalent quantity of sulphuric acid was then added, and the liberated iodine titrated in the usual way. A. G. L.

On the Determination of Hydrogen in Gas Mixtures. Francis C. Phillips. (*Journ. Amer. Chem. Soc.*, xxiii., 355.)—In burning a mixture of hydrogen and oxygen by means of palladium asbestos, it is usual to heat the tube containing the asbestos in order to prevent condensation of the water formed. If a naked flame is used for this purpose there is danger of burning hydrocarbons with the hydrogen. In the form of apparatus shown, the heating is conveniently effected by means of boiling water contained in the cup-shaped vessel *b*, *b*, 18 centimetres deep and 12 centimetres wide, which rests on three supports riveted to the inside of the brass cylinder *a*, *a*, open at both ends, 30 centimetres long and 18 centimetres wide, the rims being strengthened by wires. One of the supports is shown at *c*. The cup is silvered on the outside and the cylinder on both sides. The polished silver surfaces serve to prevent radiation of the heat to the neighbouring gas-measuring apparatus. The cup is heated by a small flame placed underneath. The tube containing the asbestos has a total length of 70 centimetres and an internal diameter of 3 millimetres. A. G. L.



Method for Preparing Strictly Tenth-Normal, Fifth-Normal, etc., Hydrochloric or Nitric Acid. Richard R. Meade. (*Journ. Amer. Chem. Soc.*, xxiii., 343.)—The author has applied his method of making standard sulphuric acid (*Journ. Amer. Chem. Soc.*, xxiii., 12) to the preparation of standard hydrochloric and nitric acid. To prepare decinormal hydrochloric acid, 12·487 grammes pure crystallized copper sulphate are dissolved in 500 c.c. of water, the copper is precipitated electrolytically, and the solution transferred to a 1-litre graduated flask. A solution containing exactly 12·215 grammes crystallized barium chloride is then added, and the solution made up to the mark; after adding 2·6 c.c. of water to make up for the volume occupied by the precipitate, the whole is mixed, allowed to settle, and the clear liquid siphoned off and filtered through a dry filter.

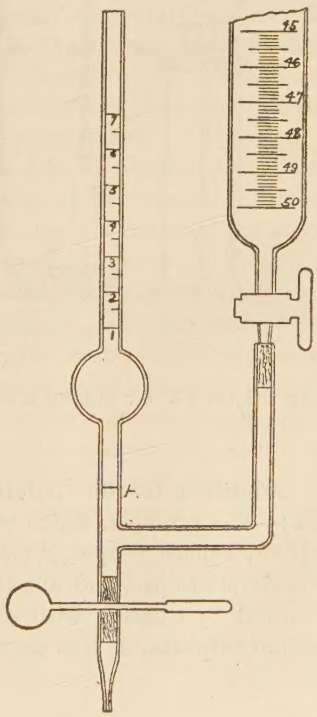
To prepare decinormal nitric acid, 13·076 grammes barium nitrate are substituted for the barium chloride. Solutions prepared in this way were found to be of exact strength and free from barium, but contained small quantities of sulphuric acid (up to 0·0320 gramme per litre). For many purposes it might be better to add a small excess of barium salt to avoid this. (*Cf.* ANALYST, xxvi., 51.) A. G. L.

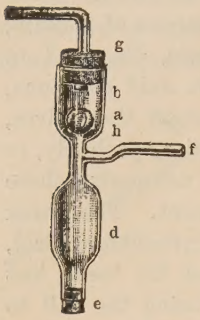
Methods of Standardizing Acid Solutions. Cyril G. Hopkins. (*Journ. Amer. Chem. Soc.*, xxiii., 727.)—The object of the investigation was to find the most exact method of standardizing solutions of hydrochloric or sulphuric acid. In the silver chloride method used, the hydrochloric acid was precipitated by a very slight excess only of silver nitrate, the precipitate filtered on a Gooch crucible and dried at 130° to 150° C. In the ammonium sulphate method excess of ammonia was added

to the sulphuric acid, the solution evaporated, and the residue dried at 120° C. Both these methods gave excellent results. For the standardization by means of sodium, the latter was weighed out under petroleum in a weighing-bottle, dissolved in alcohol, and, after diluting the solution with water, titrated with the acid solutions, using phenolphthalein as indicator. The results agreed well amongst themselves, but were higher than those obtained by the first two methods, owing, probably, to impurities in the sodium. Incidentally they provided a means of comparing these values with each other, and showed them to be in excellent agreement. The borax method, in which air-dried crystallized borax is titrated with the hydrochloric acid, using dimethyl orange as indicator, gave values which showed that the borax lost water on continued exposure to the air. This was verified by exposing the salt to air and weighing from time to time. Consequently the method is not reliable. Values obtained by electrolyzing solutions containing known weights of copper sulphate, titrating the acid liberated with sodium hydrate solution, and using this to standardize the hydrochloric acid, agreed well amongst themselves, but still were not nearly so good as those obtained by the first two methods, the maximum variation of the results found in the copper sulphate method being 0·048 milligrammes HCl in 1 c.c. solution, in the silver chloride method 0·002 milligramme HCl, and in the ammonium sulphate method 0·004 milligramme H_2SO_4 in 1 c.c. solution respectively. Consequently the author concludes that these two methods are by far the most suitable for standardizing acid solutions. A. G. L.

APPARATUS.

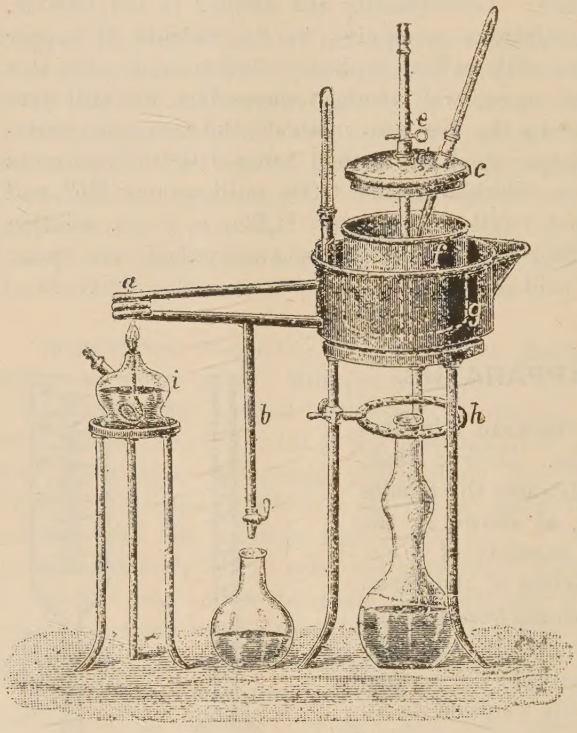
On a Modified Form of the Ostwald Burette-Calibrator. Allerton S. Cushman. (*Journ. Amer. Chem. Soc.*, xxiii., 484.)—In this calibrator the pipette has a scale engraved on its stem, as shown in the figure. The pipette must have a capacity of 2 c.c. from the mark A to about the middle of the scale. The calibration is made starting with the burette filled to the zero mark, and the pipette filled to A. The first 2 c.c. of the burette are delivered into the pipette, the reading is noted, and the pipette emptied to A; the next 2 c.c. are then introduced, and the operation is repeated down the scale. A second series of readings may be taken, starting with the burette filled to the 1 c.c. mark. Using this instrument, not a single weighing is necessary if the object is only to ascertain whether the graduations of a given burette are sufficiently uniform or not, and if absolute corrections are required it can be standardized much more easily than the ordinary form. A. G. L.





Non-return Valve for Water-Pumps. L. Wacker. (*Chem. Zeit.*, 1901, xxv., 589.)—As shown by the diagram, this apparatus consists of a glass or metal ball-valve riding on a rubber seat inside a metal or glass casing. When the pump is working properly, the valve is lifted off its seat by the suction; but should any water return, the ball falls and retains the liquid in the upper chamber, whence it eventually drops into the lower cylinder, to be withdrawn by the removal of the cork at the bottom.

F. H. L.



The Engler-Ragosine Viscosimeter. A. Ragosine. (*Chem. Zeit.*, 1901, xxv., 628.)—The author has modified the Engler viscosimeter in one or two particulars, so as to render that instrument more convenient for determinations of viscosity at somewhat elevated temperatures. As shown by the illustration, the oil-container of the apparatus is provided with a tightly-fitting cover, 5 to 8 millimetres thick, and composed of a material which conducts heat badly. At *d* it has a 1 millimetre opening to admit air into the cup. The central rod is drilled with a transverse hole, through which a pin *e* is passed to hold the rod up while oil is running out; *a* is a circulating coil of copper tube, and *b* a draw-off pipe for the water. By this arrangement the temperature of the oil will not fall more than 0·1 or 0·3° C. during a determination of viscosity at 50° C. F. H. L.

Modified Gooch Crucible. W. C. Heraeus. (*Zeits. f. angew. Chem.*, 1901, 922.)—The crucible, which was devised by Neubauer, has, instead of a perforated bottom, a piece of spongy platinum, so that no asbestos is required. Consequently, it is simple to use, and absolutely constant in weight. The precipitate is afterwards removed by treating with solvents. The modified crucible must not be used for barium sulphate, as this cannot be dissolved out of the pores of the platinum.

A. M.

REVIEWS.

PUBLIC WATER SUPPLIES. F. E. TURNEAURE and H. L. RUSSELL. New York: John Wiley and Sons. Price 5 dollars.

This book is primarily concerned with the engineering side of the question of public water supplies, but it necessarily includes a consideration of the quality of the water obtained and supplied, and this involves a chapter on the criteria provided by chemical and bacteriological methods for determining the purity of a given source. It is evident that, as the whole section touching on this topic occupies less than 100 pages in a book of more than 700, nothing elaborate or exhaustive is to be expected. But what there is is sound, plain, and sensible, and, though not likely to be new or useful to the water analyst, will serve to guide the water engineer aright in the comprehension and interpretation of the analyst's results. In this respect the work is commendable. B. B.

A MANUAL OF DETERMINATIVE BACTERIOLOGY. By FREDERICK D. CHESTER. New York: The Macmillan Company. 1901. Pp. 401. Price $2·60.

This is not a text-book on bacteriology, but a work intended to be used as an analytical key to the numerous recognised races of bacteria, which has been elaborated by the author with the praiseworthy intention of lessening the difficulties which attend the identification of these organisms. The situation with regard to the successful accomplishment of the task upon which the author has ventured is one of special difficulty, for not only is the number of recognised forms of bacteria to be dealt with very large and their descriptions widely scattered, but, far more difficult than this, the nature of many so-called "species" of bacteria is too variable, or ill-defined, to admit of exact description, and consequently of accurate identification. It is possible to overcome the first-mentioned difficulty in a satisfactory manner, and the author has accomplished this part of his task at the expense of much time and labour, with the happy result that his book contains a very useful description of some eight hundred organisms. But regarding the ill-defined nature of many so-called species of bacteria, it is impossible for any method of identification to be a *complete* success, because we have not at our disposal at present the requisite knowledge to differentiate accurately numbers of different forms of bacteria—in fact, it is, perhaps, questionable how far we can accurately use the term "species" in connection with any of the recognised bacteria. For instance, the two well-known organisms *B. coli communis* and *B. typhosus* are types possessing marked characteristics by which they may be readily identified, but between these two types there is a chain of races connecting these two ideal species, whose characteristics are so indefinite that they cannot with certainty be classed with either. It is not the fault of the book, therefore, if it does not solve such difficulties as this, for it is limited by our present want of knowledge. But, as the author proposes in difficulties such as the one mentioned, it is possible even now to arrange a system of groups in which all doubtful organisms may be placed, even if they cannot be further identified at present. We hold that the science of bacteriology with regard to the questions of species and of means for their determination must be considered as occupying a tentative position, and if the book under notice is viewed in this light we have no

hesitation in recommending it as a most useful work. We have tested it ourselves, and believe that if it is used carefully it fulfils its object of assisting in the determination of bacteria, so far as they can be determined, in a very successful manner. But here we should like to add the caution that it is essential that those who use this book should be well prepared by previous training in bacteriological methods, for bacteriology resembles all the other experimental sciences in demanding experience from its followers.

In general arrangement the book consists of two short chapters, introducing the author's systematic list of bacteria, which occupies the rest of the volume. The first chapter treats briefly on the morphology of the bacteria, and requires no special remark; but the second chapter, which deals mainly with conditions of growth, is of special interest, for in it the author has laid down the standard conditions under which bacteria should be grown, in order to study their characteristics in a systematic manner.

The author's list of bacteria is classified according to the order proposed by Migula. It may, perhaps, be satisfactory from a botanical point of view, but we do not consider the author was well advised to follow closely any botanical classification, in view of the general purpose of his book, which is to assist in determining species and not in classifying them. There is a danger under such circumstances that simplicity in determination may have to be sacrificed at times to the less important purposes of classification, and we are inclined to think that this is shown with regard to the organisms classified by the author under the genus *Mycobacterium*. These organisms, as a genus, are difficult to differentiate from the genus *Bacterium*, and we think if they had been united under one head it would have simplified and improved the author's system of determination. It is desirable, also, to call attention to another point in the book which is confusing to the reader. The manner in which the author's classified list of bacteria is introduced on page 55 is very badly chosen. This list is the main feature of the book, and occupies more than three hundred of its pages, but it commences in the middle of a page, without any special heading to call attention to its importance. These faults, to which we draw attention, and others we have noticed, are, however, of minor importance, and do not seriously impair the real value of the book, which we confidently recommend to all who have to overcome the difficulties of identifying unknown bacteria; but, as we have attempted to show in this review, the book must not be expected to act as a guide beyond the limits of our imperfect knowledge concerning the specific distinctions of these micro-organisms, and it can only be used with advantage by those who are skilled in bacteriological methods. A. J. B.

EXAMINATION UNDER THE BUREAU OF MINES AMENDMENT ACT OF BRITISH COLUMBIA, 1899.

The half-yearly examination for the qualification of Provincial Assayer, which was held at Victoria, British Columbia, on November 19, 1901, and the four subsequent days, resulted in the following gentlemen being granted licenses: D. A. Ayres, Trail; N. Colinson, Victoria; W. Stone Marshall, Duncans, and G. H. Comrie, Vancouver.

The examiners were: W. F. Robertson, provincial mineralogist, Chairman; T. Rhymer Marshall, D.Sc. (Edin.), of Glasgow; J. Cuthbert Welch, F.C.S., Chief Chemist Canadian Smelting Works, Trail; and H. Carmichael, provincial assayer, Secretary.